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Lipid Analysis of Pottery from Ayia Triada Cave, Greece: Evidence for Ritualized Consumption?

Rachel Lynn Vykukal
University of Tennessee, rvykukal@vols.utk.edu

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I am submitting herewith a dissertation written by Rachel Lynn Vykukal entitled "Lipid Analysis of Pottery from Ayia Triada Cave, Greece: Evidence for Ritualized Consumption?." I have examined the final electronic copy of this dissertation for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Doctor of Philosophy, with a major in Anthropology.

Kandace R. Hollenbach, Major Professor

We have read this dissertation and recommend its acceptance:

Erin D. Darby, Walter E. Klippel, Eleanora A. Reber

Accepted for the Council:

Dixie L. Thompson

Vice Provost and Dean of the Graduate School

(Original signatures are on file with official student records.)

Lipid Analysis of Pottery from Ayia Triada Cave, Greece: Evidence for Ritualized Consumption?

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ABSTRACT

Excavations at Ayia Triada Cave, located on the southern tip of Euboea Island, Greece, have provided evidence for the burial of at least nine individuals in the Early Bronze Age period. On the basis of intentionally smashed and arranged pottery and a thick layer of carbonized plants and animal bones, it has been suggested that feasting occurred in the cave in relation to the burials. Further evidence could lend support to this interpretation. Exceptional preservation and copious amounts of the floral and faunal material excavated from the cave suggests that the cave environment was ideal for the preservation of organic residues absorbed within the pottery as well. This study employs organic residue analysis of pottery found in the cave to investigate possible food/drink consumption and feasting practices associated with funerary rites.

Over a hundred samples of Ayia Triada pottery were collected and analyzed to determine their original organic contents. Total lipids were extracted, fractioned, and analyzed by gas chromatography and mass spectrometry, a combined technique that separates complex organic mixtures into constituent compounds for identification. These results were used to evaluate hypotheses regarding 1) the relationship between the physical food remains and the serving and storage ware; 2) possible storage of liquid goods in jars; 3) imported versus local ware usage patterns; 4) vessel homogeneity; 5) methodological concerns with respect to soil contamination; and 6) the function of culturally important sauceboats. These hypotheses helped to characterize food consumption and feasting practices associated with funerary rituals, in order to gain

access to ritual behaviors and broader cultural values of the inhabitants of Southern Euboea.

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CHAPTER ONE
INTRODUCTION

While many features of a burial can be relatively straight forward to investigate within the archaeological record, the ritual behavior surrounding an inhumation can be difficult to ascertain. For example, skeletal remains can be analyzed to determine age and sex of the individual, pathologies can be determined to assess disease and overall health, and grave goods can be used to investigate the associations between sex and status. However, funerary rituals remain one of the most challenging aspects of a burial to reconstruct, especially in early societies where written accounts do not exist. It is likely that structured patterns of behavior accompanied burials, but the evidence usually does not survive and often we are left to only speculate about the nature of those rituals. From time to time, however, the archaeological record provides a wider glimpse into ritual behavior. Ayia Triada Cave on the Greek island of Euboea is one such case, where evidence for ritualized feasting in connection with human burials was uncovered dating to the Early Bronze Age.

Excavated between 2008 and 2010, Ayia Triada Cave was occupied in the Late and Final Neolithic (5300-3200 BC) and Early Bronze Age II [EBA II] (2650-2250 BC) periods (Mavridis and Tankosić 2009a, 2016a, 2016b). There is no evidence to suggest that the cave was ever used for habitation, likely due to its cramped and narrow layout. The interior of the cave could not have held many people at once, especially in the East Chamber where EBA II deposits were found (Mavridis and Tankosić 2016a:214). Evidence suggests that at least nine individuals were buried in the cave in EBA II,

although the incomplete and fragmentary nature of the skeletal material precludes any determination of simultaneity (Mavridis and Tankosić 2016a:224; Žarko Tankosić, personal communication 2017). The human remains were found atop a layer of burnt macrobotanical and faunal remains, including grains, figs, pulses, and sheep/goat bones (Mavridis and Tankosić 2016a:223). Serving and storage vessels were found in large numbers, including bowls, storage jars, jugs, cups, and sauceboats, some of which were imports of Cycladic or mainland Helladic origin (Mavridis and Tankosić 2016a). It appears that fine ware was intentionally placed below the human remains, while coarser pottery was placed at the edge of the remains (Mavridis and Tankosić 2016a). Many of the coarse-ware jars appear to have been broken *in situ (*Mavridis and Tankosić 2016a). DNA analysis of the skeletal remains is being conducted to determine kinship associations between the individuals, while strontium isotope and stable oxygen isotope analyses will investigate geographic origins (Mavridis and Tankosić 2016a).

Mavridis and Tankosić (2016a) suggested that feasting occurred in the cave in connection with burials, but the details remain as of yet unstudied. Ayia Triada Cave provides a unique opportunity to explore behaviors surrounding the consumption of food in burial contexts in EBA II, because of its excellent preservation and undisturbed contexts. Here I investigate food consumption in burial contexts through chemical residue analysis of the pottery found in the cave. I analyzed pottery samples to determine their original contents using gas chromatography and mass spectrometry (GC/MS), a technique that separates complex organic mixtures into their constituent compounds for identification. This method can aid in determining if these vessels reflect highly specialized consumption or if they align more closely with everyday domestic patterns,

which in turn can shed light on the community to which the deceased and the participants belonged.

Statement of the Problem

Doumas (1977) commented over forty years ago, and it remains true today, that we know very little about rituals in Early Bronze Age Greece. A primary reason for this lacuna is that countless EBA burials from the Cycladic islands have been looted and contexts destroyed by people in search of prized marble figurines and other grave goods (Broodbank 2008; Doumas 1977). If we turn our attention to the island of Euboea and its southern region, we know even less about burial traditions. Although extensive surface surveys have been carried out through the Southern Euboea Exploration Project, only limited excavations have been conducted (Cullen et al. 2011; Cullen et al. 2013). Of particular importance to this study, no settlements or cemeteries dating to EBA II have been fully excavated on the island. We know relatively few details about the daily lives of inhabitants of Southern Euboea in this early period and even less about their rituals. Until the last century, Euboea was thought to have been uninhabited for most of its history, despite the fact that the island is relatively close to the mainland at its central point and is within a few hours of maritime travel to other islands (Broodbank 2000; Cullen et al. 2013).

In addition to the lack of information about EBA rituals, diet and foodway studies focusing on this period are non-existent at many sites. In particular, Megaloudi (2006) points out how sparse the paleoethnobotanical record is for most of the Bronze Age, which can be attributed to biased excavation methods and/or poor preservation of remains

in the temperate environment. Ayia Triada has an unusually large cache of macrobotanical remains preserved (Mavridis and Tankosić 2016a:223). However, these remains, as is the nature of macrobotanical remains in general, can only reveal part of the story of was consumed and stored in the cave. There are many substances that only leave ephemeral traces in the archaeological record, if they leave traces at all. Herbs, leafy greens, roots, and tubers do not preserve well, nor do liquid concoctions, such as fermented beverages or oils. These foodstuffs must be investigated in another way, which is precisely the role chemical residue analysis can play in foodways studies. Chemical residue analysis of organic compounds can offer a wealth of new information to supplement the palaeoethnobotany and zooarchaeological record.

Organic residue analysis has not been employed regularly in research focusing on prehistory of the Aegean. When occasional studies have applied this methodology, they have mostly focused on Minoan Crete or on identifying specific foods/drinks (e.g. Tzedakis et al. 2008). It is unfortunate that an entire layer of data often remains untapped and ignored in Greek archaeology. This is one of the few studies of this period to employ a systematic program of organic residue analysis to answer anthropological questions.

Theoretical Approach

I approached this research within the context of practice theory. This theoretical framework views individuals as organizing their world within a set of sociocultural schemes that they create, reproduce, and renegotiate (Bourdieu 1977). Behavior is governed, but not precisely dictated by these systems of schemes or *habitus* (Bourdieu 1977:73). Actors can "generate an infinity of practices adapted to endlessly changing

situations, without these schemes ever being constituted as explicit principles" (Bourdieu 1977:16). In other words, individuals operate within an arbitrary system, but they can act according to their individual desires. Although arbitrary, the system is seen as the natural order by society; Bourdieu (1977) termed this collective perspective *doxa*. This approach emphasizes the adaptability and creativity of social actors, rather than casting them as static cogs of a larger system (Dietler and Herbich 1998). There is a reciprocal relationship between actors and the social structures; the interaction is active and transformative. *Habitus* is initially constructed, but "reproduced as networks of practice that stretch over time/space" (Barrett 1991:4). Practices related to food consumption and burial rituals intersect within the arena of funerary feasting, which can be explored in terms of practice theory.

The study of foodways abounds with areas in which to access *habitus* of past societies, from the production to the consumption to the disposal of food and all phases in between (Goody 1982:37). Food is laden with meaning and by analyzing its systems, we can understand how a culture organizes and understands its world (Douglas 1966, 1975). Food embodies "a protocol of usages, situations, and behavior" (Barthes 1997:21). It communicates and signifies aspects of social structure and can be reinforced or redefined by those consuming it (Barthes 1997:21-22). Practices surrounding foodways are repeated daily, which become habits that are shared collectively and yet experienced by the individual. An example of an adaptable consumption pattern is the British 'meat and two vegetables' structure of daily meals; this structure is essentially replicated even at meals of greater importance, such as holiday meals, where it is merely elaborated upon (Douglas 1972; Douglas and Nicod 1974). Douglas and Nicod were structuralists, but if

we infuse this example with the social actor's ability to adapt practices within their own experiential realm, practice theory emerges.

Feasting is a specific arena within food consumption and is defined in various ways by scholars. Dietler and Hayden's (2001) volume on feasting provides a handful of definitions alone. To illustrate this point, Hayden (2001:28) defined feasting as the consumption of a meal of unusual foods by two or more people for a special occasion. In the same volume, while Dietler (2001:65) emphasized the shared consumption of food for an occasion, but not necessarily that special foods must be consumed or that the 'ritual' be overly elaborate or sacred in nature. Feasts are the "marked form to the unmarked meal" (Dietler 2001:69-70). In the archaeological record, it can be difficult to identify feasting activity, especially if the feast entails a group of less than 50 people (Hayden 2001:47). Often, it is only a distinctive location of the food consumption that signifies feasting activity occurred (Hayden 2001:39-40). Regardless of how one defines it as a term or in the archaeological record, the goal of feasting is a social one to establish or reaffirm bonds between individuals or groups and to foster cohesion within participants (Dietler 2001; Hayden 2001:9). Feasting moves beyond the biological need to ingest food for survival.

Hamilakis and Sherratt (2012) discussed feasts as mnemonic devices, an idea that is not new. It has been theorized within sociology and cultural anthropology. One such example is Lupton's (1994) study on the intersection of food and memory. She collected solicited but not structured food memories from a group of undergraduate and graduate students. This group of students crossed all ages, classes, and genders. However, it was striking how many of the memories shared a common theme: the majority of memories

brought up food in relation to social relationships. The crucial element was not necessarily what was eaten, but the social contexts in which it was eaten. The consumption of food is an act of embodiment (Dietler 2001:72).

Hayden's (2001) work has been instrumental in defining the types of feasting and the practical social benefits they may bring to the host(s), be it an individual, a family, or a community. Feasts can be analyzed on many levels, the most basic of which is in terms of their form and their function (Hayden 2001:36-37). The form would be related to the emic category of the feast: marriage feast, maturation feast, harvest feast, funerary feast, etc. (Hayden 2001:36-37). The function or benefit of feasting may be to summon a labor force, create relationships for marriage or alliance, gain status, or even to exclude groups, to name a few (Hayden 2001:37-38). A feast may utilize multiple roles at any given time and the various participants may engage in feasting for different functions (Hayden 2001:36). Hayden (2001:38) outlined three main functions under which all feasts fall: those that are for alliance and cooperation, those that are for economic gain, and those that are for status display. Dietler (2001:76-86) also identified three types of feasts, these all grounded in sociopolitical power: diacritical, empowering, and patron-role. Diacritical feasts are conceptualized in the same manner as Hayden's (2001) feasts for display; they are meant to underscore social distinctions with categorically differentiated food (Dietler 2001:85-86). Empowering feasts can be used for "negotiating social positioning" in such a way that draws strength from a "collective misrecognition and euphemization" between the participating groups (Dietler 2001:76). Patron-role feasts work to portray and reinforce one-sided power relationships of host to patron via for redistribution of surplus (Dietler 2001:82-83).

Burial rituals, much like feasting, are at their core social endeavors. Rituals are comprised of action as the basic unit of analysis (Barrett 1996:396). The performance of mortuary rituals involves a series of actions and practices by individuals (Barrett 1996:396). Social cohesion is reinforced through burial practices after a cultural disturbance like death (de Martino 2000). Mortuary rituals are performed for the benefit of the living and serve vital functions within the community (Laneri 2007). They create distance between the living and the dead and create a structure for the memory of the latter. They reproduce, expand, and reconstruct various social identities of the living and the deceased (Laneri 2007:5). The social and cultural identity of the deceased is condensed and often transformed into that which the family or community desires to ascribe to the individual in death (Chesson 2007). Lastly, burial rituals magnify mutually held beliefs to reinforce the structure of the community (Laneri 2007:5).

The tangible elements of the rituals encompassing death serve as the "focal point in the social and mnemonic landscape of the society" (Laneri 2007:4). The memorializing of the dead is "strongly emphasized by the disposal and/or destruction of material culture" (Laneri 2007:8). They are experiences that are transformative. The active experience of ritual allows people to overcome disruption of death and regain a sense of stability (Laneri 2007:6). Although the rituals are performed from a place of common beliefs, every individual interprets them through their own lens of experience (Barrett 1991:5,7).

Research Plan

In order to investigate feasting practices associated with funerary rituals, I sampled pottery from the EBA II layers in the cave, those that were associated with the human burials, grave goods, and burnt organic remains. The goal of my sampling strategy was to be systematic. I sampled a total of 115 pottery sherds that belong to both local and non-local types. I collected pottery samples from storage jars, bowls of assorted sizes, sauceboats, a *pyxis* (lidded vessel), and an open vessel with a handle. The remaining sherds were from unknown vessel types. The sherds were selected from all fabric types: fine, medium-coarse, and coarse wares.

Several scenarios could emerge from the residue data and each would imply a separate set of meanings and behavior. Firstly, the vessels deposited deep in the cave could be empty, unused pottery, possibly created specifically to be deposited with the dead. These would all contain either no residues or residues below the threshold for interpretation. Secondly, the pottery assemblage could display a narrow range of foodstuffs that were reserved for burial feasting and reflect highly specialized consumption. These specialized foods could be imported foodstuffs from other regions or merely foods that were scarcer or more labor-intensive to produce (Hayden 2001:40). A third and final scenario is that the pottery assemblage could be comprised of intensively used vessels that contain a range of presumably everyday foodstuffs.

To investigate how well the pottery and its contents stack up against these situations, I evaluate a series of hypotheses using the organic residue data. These hypotheses address the variability of organic contents across the sampled pottery,

investigate the link between vessel contents and vessel types, and assess the validity of some methodological checks for soil contamination.

- *Hypothesis 1: The plants identified in the burnt organic layer were served or stored in the vessels deposited in the cave.*

  I expect to find either the biomarkers for these plants in the serving vessels as mixtures or as single sources, or I would expect to find that the storage jars had negligible quantities of residues, which would suggest that they held dried goods. It is not expected that lipids from dried goods, particularly plant foods, would infiltrate the vessel walls.

- *Hypothesis 2: The constricted-neck storage jars, the ovoid and bulbous types, held single commodity liquids, such as olive oil or alcoholic beverages. The liquid storage vessels would have been sealed in some manner to waterproof their interiors and prevent liquid loss.*

  I expect to see residues that contain biomarkers associated with oils and/or alcoholic beverages. I anticipate that the remains of a sealant will be extracted from the vessel walls, such as plant resin, pitch, tar, or beeswax in these jars.

- *Hypothesis 3: There is homogeneity of use within pottery types. In other words, different pottery types were reserved for storing or serving specific foodstuffs.*

I predict that each vessel type, storage jars, bowls, sauceboats, etc. will contain residues with relatively similar quantities and compounds that generally represent the same foodstuffs. In contrast, I expect to see differences between vessel types.

- *Hypothesis 4: The organic contents of imported vessels can be differentiated from those of local vessels, implying specialized use of the imported pottery types.*

  I expect to find differences that may reflect imported foods, scarcer commodities, items that are more labor intensive to produce, or even vessels with a narrower range of plant and animal resources than the local types. A range of characteristics could set the imported vessels apart from the local types.

- *Hypothesis 5: Handles can be used to test soil contamination, which is particularly useful in the absence of soil samples.*

  The handles should display a low lipid content overall, likely below the threshold for interpretable residues, particularly if there is no soil contamination. These lipid profiles should indicate background contamination from the soil that might influence the other sample residues.

- *Hypothesis 6: Sauceboats were used for serving liquids and were connected to ritualized drinking practices.*

  I expect to find evidence for beverages, such as wine or other fermented products, and a relatively even distribution of lipids across the vessel surface. The latter is anticipated since heat probably was not applied to these vessels that would

preferentially destroy lipids and/or create lipid distribution patterns associated with boiling/cooking.

Summary

Chemical residue analysis can provide complementary evidence to plant and animal remains, which often have taphonomy and/or recovery issues. I am examining organic residues from a range of vessel types and sherds at Ayia Triada, an EBA II site—a time and place where there is relatively little information on foodways. Through the lens of practice theory, I explore feasting and funerary practices using residue analysis.

In Chapter Two, I contextualize Ayia Triada in its regional and temporal landscape in the first half and then focus on foodways and burial customs of the prehistoric Aegeans in the second half. The next three chapters delve into organic residue analysis. I discuss the methodological underpinnings of organic residue analysis and explain the specific methods employed in this study in Chapter Three. In Chapter Four, I present the results of the residue analysis of Ayia Triada pottery, followed by an in-depth discussion of these results in Chapter Five. I conclude with the implications of this research for the practice of feasting in ritual contexts.

CHAPTER TWO
CHRONOLOGICAL AND REGIONAL OVERVIEW

Calligas (1984:89) remarked that Euboea is a "melting pot" between the Helladic mainland and Cycladic islander worlds. Even though Euboea is an island, it is geographically close enough to the mainland and nearby islands to have interacted with and been influenced by various cultural groups. Therefore, in order to understand Ayia Triada, the site must be contextualized within the greater region, which includes southern and central mainland Greece (Attica, Boeotia, Corinthia, and the Argolid), the coastal islands of Aegina, Euboea and Skyros, and the island group of the Cyclades. In this chapter, I provide a historical overview of the periods leading up to the Early Bronze Age, starting with the Late Neolithic II/Final Neolithic, where we begin to see the emergence of Bronze Age society. I follow with a diachronic and regional survey of diet and burial practices. Finally, I discuss findings from the Ayia Triada excavation and the Southern Euboean landscape to which it belongs.

Chronology of Prehistoric Greece

An exhaustive review of the debates within Aegean chronology is beyond the scope of this research, as there is still considerable disagreement amongst scholars. Many excellent reviews have already been devoted to this topic (e.g. Broodbank 2000; Manning 1995, 2010; Phelps 2004). One underlying reason for contention among scholars is that Greece in prehistory was never a "monolithic cultural entity" (Tankosić 2011:24). Greece is a fragmented landscape geographically, which contributed to its cultural fragmentation

throughout prehistory. Relative chronologies have usually been based on local developments within ceramic sequences, which have not been easily reconciled in relation to one another. Compounding the problem is that not all areas have well stratified sites from all time periods. A brief overview will suffice here.

Early scholars of Greek prehistory divided Greece into regions and applied cultural labels for inhabitants who lived in these regions: Helladic for the mainland, Cycladic for the cultures of the eponymous archipelago, and Minoan for the culture that thrived on Crete (Shelmerdine 2008:3). The Bronze Age was then divided into Early, Middle, and Late periods each with three subdivisions per region (Shelmerdine 2008:3). The Early Bronze Age of the mainland was subdivided into Early Helladic I, II, and III (EH I-III) periods based on excavations at Korakou and at Eutresis in the early twentieth century and later clarified through the Lerna excavations in the 1950s (Blegen 1921; Caskey 1960; Goldman 1931). However, C. Renfrew (1972:53) later proposed designations for distinct assemblages of material culture, burial customs, and the sites they were associated with, which he argued were "in effect cultures in the well-defined archaeological sense of the term" and which could be used in addition to traditional periodization naming (EH I-III). This was meant to supersede strictly chronological periodization, because Renfrew argued that the assemblages could be contemporaneous (C. Renfrew 1972:53). For the Cyclades in the Early Bronze Age, he coined the cultural group names of Grotta-Pelos, Kampos, Keros-Syros, Kastri, and Phylakopi I and for the mainland, Korakou and Eutresis, based on Blegen and Goldman's excavations of the sites with the same name (C. Renfrew 1972:53). C. Renfrew (1972:76) lumped both the mainland and the Cyclades into the same cultural unit he called Attica-Kephala for the

early Final Neolithic, for reasons which will become apparent in the historical overview. Grotta-Pelos emerged in the late Final Neolithic and extended until the end of Early Bronze Age I (Broodbank 2000:53; C. Renfrew 1972:53).

Doumas (1977) later followed Renfrew's convention for the Cycladic material, because still not enough Cycladic settlements had been excavated to produce and compare long stratigraphic sequences (Broodbank 2008; Manning 2010; Renfrew 2010). Furthermore, most of the evidence from the Cyclades derived from cemeteries, which were heavily disturbed by looting and, therefore, had incomplete stratigraphic records (Doumas 1977). Since then, researchers have either continued in this same vein with the cultural group names, subscribed to the EC/EH chronologies and then subdivided further into ultra-specific subperiods, or crafted site specific micro-scale chronologies (i.e. at Phylakopi and Lerna). Similar to the confusing naming systems of the region in EBA, the last stage of the Neolithic is named differently according to which scholar and site one is referencing. Mavridis and Tankosić (2016b) provided an excellent overview of the various naming systems. The dizzying amount of relative chronologies and naming systems have often muddled the actual changes they were meant to represent in the Bronze Age and the periods leading up to it. Manning (2010:11) astutely observed, "Chronology has become both framework and constraint, friend and problem."

It is now apparent that both the Cycladic and mainland cultural groups roughly fall into the tripartite chronological descriptions of Early Cycladic I, II, and III and Early Helladic I, II, and III for the mainland with a couple of minor transitional groups in between (Table 2.1). To reflect how Euboea is neither solely Cycladic nor Helladic and to align this study with the previous studies of Ayia Triada, I use the Early Bronze Age (EBA) I,

II, III designations that correspond to EC I-III and EH I-III. I employ the more encompassing Late Neolithic I and Late Neolithic II/Final Neolithic (LN II/FN) terminology for the period preceding EBA for the same reasons of alignment with earlier work.

Colonization of the Islands

Cherry (1981, 1985) has written extensively on patterns of colonization of the Aegean islands. He disputed Evans' (1977) earlier claims that the islands were initially settled in the Early Neolithic, because of the emergence of farming. Crete and Cyprus, the largest islands in the Eastern Mediterranean region, were settled in the Early Neolithic, which Cherry (1981:52) argued represented purposeful, directional colonization. However, it was not until the Late Neolithic, one to two millennia later, that we see the first permanent settlements outside of Crete and Cyprus on the large littoral islands, such as Euboea, Kea, and Aegina (Cherry 1981:52, 1985:17). These islands were settled next, because inhabitants could use the mainland as a buffer in the event of catastrophes. Next, most of the larger non-littoral islands and many of the smaller islands, most over 100 km from the mainland, were settled relatively quickly within the Bronze Age (Cherry 1981:52). The Early Bronze Age was a time of settlement of the far smaller islands with small communities (Cherry 1981:52). Less than 20% of the Aegean islands were settled before the Bronze Age, which is in stark comparison to the roughly 70% that were settled by the end of the middle part of EBA (Cherry 1981:52, 1985:18).

The key to this successful settlement was exchange and mobility via seafaring,

Table 2.1 Absolute and relative chronology of the Late and Final Neolithic and Early Bronze Age Periods with Ayia Triada's occupation noted (Coleman 1992, Manning 2010, Mavridis and Tankosić 2009a, 2016a, 2016b; C. Renfrew 1972)

Dates (BC)	Relative Chronology	Cyclades	Sub-period dates of Cyclades	Mainland	Sub-period dates of Mainland	Ayia Triada
5300-4300/4200	LN I					X
4300/4200-3200	LN II/FN	Saliagos	4300-3700			X
		Attica-Kephala	3300-3200	Attica-Kephala		
3100-2650	EBA I	Grotta-Pelos	3100-3000	Korakou		
		Kampos	2900-2650			
2650-2250	EBA IIA	Keros-Syros	2650-2500	Eutresis		X
	EBA IIB	Kastri	2500-2250	Lefkandi I	2500-2200	
	EBA III	Phylakopi I		Tiryns		

harkened by the development of longboats (Broodbank 2000; Cherry 1981,1985). These small island communities of the EBA could rely on nearby islands for exchange of marriage partners and goods in times of need. Cherry (1981:59) argued that the risks of living on the small Aegean islands were high, because the environment was fragile and unpredictable. Halstead and O'Shea (1982) argued that there is an inverse relationship between arable land and inter-annual crop variability. Risks were mitigated by island populations through strategies such as maintenance of exchange networks, mobility, diversification of crops to protect against crop failure, and the wider exploitation of wild resources (Cherry 1981; Halstead 1981). Cherry (1981:60) argued that we should think of Neolithic colonization as "many, tentative, impermanent, short-distance reciprocal movements by mere handfuls of individuals" carried by boats loaded with domesticated animals. It is important to note that seafaring in the region had been known several millennia before the permanent settlement of the Aegean islands, as evidenced initially by obsidian that originated from the island of Melos discovered in Paleolithic and Mesolithic layers of mainland Franchthi Cave (Jacobsen 1969,1999; Perlès 1987; Renfew and Aspinall 1990). More recently, Paleolithic stone tools have been found on several Greek islands that were not connected to the mainland at the time, e.g. Naxos, Crete, Zakynthos (Broodbank 2000; Cherry 1981; Runnels 2014 and references therein). Since no Paleolithic habitation sites have been found, it is unknown whether they represent temporary visits or longer-term habitation (Runnels 2014:222).

In biology, the founder effect states that founder populations quickly diverge genetically from the original population, because they only carry with them a small proportion of the gene pool from which they came. Cherry (1985) applied this concept to

explain cultural 'founder' populations in the Aegean. The founder populations cannot necessarily reproduce all aspects of their culture, because they carry with them only a small part of the larger society's technological know-how and cultural traditions. The result is that island founder cultures diverge quickly from their parent cultures and the differences become even more apparent with isolation (Cherry 1985:26). He argued that these cultural mechanics could explain why Cypriot and Cretan material cultures were so different from other parts of eastern Mediterranean, as well as why the first phase of the LN Cycladic culture, the Saliagos culture, was so different from the mainland and other areas (Cherry 1985:26-27).

Late Neolithic II/Final Neolithic (4300/4200-3200 BC)

The region encompassing Central and Southern Greece, Euboea, and the Cyclades is linked by shared assemblages and cultural practices in the LN II/FN. This led Renfrew to attribute the sites to the Attica-Kephala cultural sphere (C. Renfrew 1972:75-76). The LN II/FN landscape seems to be a dispersed network of small sites made up of single dwellings or hamlets across the islands and the mainland (Tomkins 2010:40). Beginning in the Late Neolithic, people began to settle marginal environments, those that are considered agriculturally less productive (Tomkins 2010:39). Most of the Cyclades can be characterized as marginal with their rocky, thinly soiled landscapes and small patches of fertile soils prone to erosion (Broodbank 2008:47). Caves start to be used more frequently in the LN II/FN (Kouka 2008:272). These settlement patterns contributed to the need for trading in an effort to mitigate the risks of living in marginal environments (Tomkins 2010). In association with this move into more marginal areas, the LN II/FN is

characterized by a trend towards more isolated households that function as individual socio-economic units, free from communal obligations with a greater independence to accumulate wealth on their own (Tomkins 2010:39, 42). These households were larger than the nuclear family (Tomkins 2010:39). In some areas of the mainland, cooking installations moved indoors or behind closed yards, in comparison to the communal cooking spaces of the earlier Late Neolithic (Halstead 1989:77). It is difficult to reconstruct the social structure of the LN II/FN, because of limited evidence. Society was probably roughly egalitarian, but these trends suggest that household interests began to be pursued over communal interests at least to some extent (Tomkins 2010:40-42).

Reasons for the shift towards settling in marginal environments are debated. Sherratt (1981) argued that marginal areas were settled in part because of the secondary products revolution enabled it. People began to employ traction animals to cultivate agriculturally poor areas; thus, more land was cleared that allowed larger herds to be kept and spurred their long-term use for secondary products, which could then be traded (Sherratt 1981). Halstead (2008) criticized the link between the secondary products revolution and marginal colonization. He argued instead that it was the result of dispersion of settlement patterns and an increased emphasis on individual households, instead of the community, that triggered the colonization of marginal areas. This allowed individual households the freedom to accumulate a surplus that could be used to initiate and maintain interregional exchange networks and thereby, enhance their own economic survival (Halstead 2008:248).

Maritime movement increased around the Aegean in LN II/FN, likely initiated by the invention of the longboat and further encouraged by the desire to trade prestige items

(Tomkins 2010:41). People were probably trading in low volumes, but the goal of trade was more social than economic; building relationships was key to gaining access to resources and marriage partners (Tomkins 2010:41). Obsidian from Melos in the Southern Cyclades was being traded. People developed copper metallurgy in this period as the first form of metalworking in the Aegean; small trinkets and heavy tools were the most common items made (Nakou 1995:4; Renfrew 2010:86; Tomkins 2010:41). Metal objects were found mostly in settlements and caves instead of burials in this period (Nakou 1995:6-7). Several sites on Kea have evidence for metallurgy with both the finished products and their byproducts (Coleman 1977; Nakou 1995:5-6). Connections with the Balkans are evident with the metals of central and southern Greece (Nakou 1995:6).

On the mainland, settlement patterns are understood mostly from surface surveys, since there are no well-stratified LN II/FN sites (Pullen 2008:20). Surface surveys reveal a proliferation of sites and a diversification of site locales (Pullen 2008:20-21). There was an increased emphasis on pastoralism and an expansion into slopes and uplands (Pullen 2008:20-21).

In the Cyclades, the islands were thinly settled overall and LN II/FN architectural remains are scarce (Broodbank 2008). Settlements start to shift towards the end of this period from the earlier Neolithic patterns of nucleated settlements to smaller communities spread inconsistently across the landscape (Barber 1987; Broodbank 2008:52). Kephala on the island of Kea is an example of one of these small LN II/FN settlements, with stone structures, a well-preserved cemetery of stone-built cist graves, and an estimated population of c. 50 people (Coleman 1977). Architectural remains from this phase have

also been found on Akrotiri on Santorini, but are heavily obscured by the overlaying later periods (Sotirakopoulou 1990).

The well-preserved, large site of Strofilas on Andros is the exception to the rule and is changing our understanding of LN II/FN settlements. Strofilas is located on a naturally defensive spot between two harbors and is inaccessible from the coast by sheer cliffs (Televantou 2008:44). The settlement featured a system of the earliest fortification walls in the Aegean, positioned to prevent inland access (Televantou 2008: 45). Prior to the excavation of this site, fortification walls were thought to have first been constructed in the EBA II. For the first time in the Cyclades, a settlement was chosen for its defensibility (Broodbank 2008:52). Well-preserved architectural remains consist of rectangular and apsidal structures, some of which may have held a second story, and a possible sanctuary, rare elsewhere in the Cyclades (Televantou 2008:45). Ring idol pictographs carved in bedrock and ring idol pendants were found, linking the site ideologically to the rest of the Aegean world (Televantou 2008:49). The ring idol is an outline of a circle with a protrusion at one end that may have held religious significance (Televantou 2008:49 and references therein). Monumental rock art has been found at Strofilas, showing agricultural, hunting, and maritime activities with longboats, canoes, and animal cargo (Televantou 2008: 47). This was the first depiction of boats in the prehistoric Aegean.

Pottery of the LN II/FN is marked by an increase in the number of coarse-ware vessels, reaching 95-100% of the pottery assemblages at some sites (Perlès and Vitelli 1999:99). The emphasis of pottery is its use in the "ordinary domestic sphere" with bowls and jars being most common (Perlès and Vitelli 1999:105). Large storage vessels like the

pithoi found at Kephala remained common in this period (Coleman 1977). This can be seen as a continuation of the increase in storage pottery initiated in the preceding Late Neolithic I period (Cavanagh 2007:117). Pottery in the Cyclades and Attica is similar with red and burnished ware decorated with pattern burnishing, incising, and grooved surfaces (Sampson 1993b:293-292; C. Renfrew 1972:75-76; Renfrew 2010:86). Rolled rim bowls and 'cheese pots' appeared towards the end of the LN II/FN period; the latter are perforated basin-like vessels called as such because they resemble modern pots used to make cheese (Bogucki 1984; C. Renfrew 1972:155; Sampson 1981, 1993b). Scoops are typical across the Aegean in this period and are likely ritual in nature (Coleman 1977:16-17; Perlès and Vitelli 1999:105).

Early Bronze Age I (3100-2650 BC)

The evidence for EBA I is scarce, usually based solely on a small amount of pottery at any given site (Kouka 2008:275). Many sites are only scantily published, at least from the early part of EBA I. Because of these factors, the period is generally not well defined across any of Southern, Central, or Cycladic Greece. The evidence we do have points to continuity from the previous period, based on a handful of archaeological sites on the mainland and the Cyclades. On the mainland, archaeological evidence comes mostly from settlements, such as Eutresis in Boetia and Tsoungiza in the Argolid. These are associated with the Korakou culture (C. Renfrew 1972:53). In EBA I, the population increased along with the number and range of sites, especially in coastal Attica, while sites in higher elevations decreased (Kouka 2008:275; Pullen 2008:22). Settlements were newly founded near the Laurion mines on the Attic coast (Kouka 2008:275). Other sites

in Attica and Euboea were established, e.g. Ayios Kosmas, Zagani, and Manika, and were connected with already established sites (like Tsepi) into the short-range trade network of silver and other metal ores (Kouka 2008:275). Not only were Cycladic artifacts recovered from these sites, but they also had Cycladic style cemeteries (Kouka 2008:275). Burial traditions are discussed in detail in the next section. A shift away from pastoralism and towards agriculture is evidenced on the mainland (Pullen 2008:22). Extensive trading networks existed in EBA I and were likely incited by settling in more coastal areas on the mainland (Forsen 2010:61; Pullen 2008:24). Although we have a good understanding of settlement patterns overall, the details of the period's architecture remain a mystery, since much of the EBA I remains still lie under later occupations that are difficult to access (Pullen 2008:24). The scant architecture that has been found includes wall remains and a room at Eutresis, a cistern and a few pits at Tsoungiza, and walls from Perachora, Vougliameni (Caskey and Caskey 1960; Fossey 1969; Pullen and Allen 2011). Spindle whorls provide evidence for textile production (Pullen 2008:24).

In contrast to the mainland, there are no settlements dating to the EBA I period in the Cyclades, but there are a handful of cist grave cemeteries on Melos, Paros, Naxos, and Siphnos (Renfrew 2010:87). These are attributed to the Grotta-Pelos culture of the Southern Cyclades, which emerged first in LN II/FN (Renfrew 2010:87). Broodbank (2008:53) remarked that it is difficult to define this period in the Northern Cyclades, however. The lack of the EBA I architecture in the Cyclades could be the result of a shift towards constructing houses with perishable materials (Barber 1987:46; Doumas 1977:13). There is little evidence for the use of the plow, which is not surprising given the rocky terrain of the Cycladic islands (Broodbank 2008:53). The number of cemeteries

increased in EBA I on the Cyclades, which likely indicates a corresponding increase in the settlements (Barber 1987:133; Doumas 1977:14). Obsidian blades, marble figurines, as well as marble and ceramic vessels were found in burials across the Cyclades (Broodbank 2008:60; Doumas 1977:60). Early in this period, traces of metalworking were found at settlements and at the ore sources, such as at Siphnos, but the metallurgical products are surprisingly absent in the archaeological record (Nakou 1995:8; Wagner et al. 1980:76). However, in late EBA I and continuing into EBA II, metal began to be deposited with the dead, a clear shift from its depositional patterns in the previous period (Nakou 1995:7). The trend toward small, dispersed settlements initiated in LN II/FN continues into EBA. These settlements would have held one to a few families, as evidenced by the size of most of the cemeteries with between 15-20 people (Barber 1987:134; Broodbank 2008:54). Trade is evidenced on the mainland, with the aforementioned Cycladic grave goods and funerary architecture in Attica. There is support for longer range interactions between the Cyclades and elsewhere in the Aegean, such as the Cycladic colony or "gateway community" on Crete at Hagia Photia (Betancourt 2008:240). The social structure was likely kin-based, as evidenced by the elaborate graves (Tankosić 2011:49).

Pottery from the Grotta-Pelos culture of the Cyclades is brown to black burnished with a thick, gritty fabric and often with incised decoration filled with white paste; the herringbone design is a common motif (Barber 1987:89; C. Renfrew 1972:153-155; 2010:87). Common shapes are rolled rim bowls with lugs, bowls with T-shaped rims, collared jars, cheese pots, and cylindrical or spherical lidded boxes, known as *pyxides* (singular *pyxis*) (C. Renfrew 1972:152-169; Sampson 1993a:30-44). Marble vessels were

also used, such as open bowls, flat beakers, and footed jars (C. Renfrew 1972:159, 2010:86).

The pottery of the mainland is not that different from the pottery of the LN II/FN and often cannot be more precisely dated than FN/EBA; unslipped or red-slipped, burnished, and often incised pottery continued into this period (Forsen 2010:53; Pullen 1995:11-12; C. Renfrew 1972:100). Other decorations persisted, such as the taenia band of aligned finger impressions and *kerbschnitt* pattern of alternating impressed triangles (Pullen 1995:10-19). Characteristic shapes are bowls, deep and hemispheric or with incurving rims, *askoi* (vessels shaped usually like an animal used to pour small amounts of liquid), one handled cups or jugs, and cooking pots with ridging near the rim (Caskey and Caskey 1960; Cullen et al. 2013:72; Pullen 1995:10-19).

In the transition between ECI and ECII, pottery referred to as the Kampos group emerged with a wide range of shapes (Broodbank 2008:60). Characteristic vessels are wide, pedestaled bowls commonly referred to as fruitstands, bottle-shaped vases, collared jars, deep bowls or cups with incurving walls, cylindrical, conical, or biconical pyxides, and a distinctive type of "frying pan" (Coleman 1985:196-197; Doumas 1977:18-20; Karantzali 2008:243-252; Renfrew 1984:50, 2010:87). The latter is a round, shallow basin of unknown function with handles on one side and usually incised or impressed designs on the bottom and sometimes the sides (Coleman 1985:193; Doumas 1977:18-20; Renfrew 1984:50,2010:87). The Kampos frying pans had elaborate incised decorations and mostly barred handles (Broodbank 2008:60; Coleman 1985:196-197; Renfrew 1984:47). Frying pans are found often in funerary contexts in the Cyclades, but are found in both settlements and tombs on the mainland, although this could be a result of the

scarceness of known settlements in the Cyclades (Coleman 1985:197-200; Pullen 2008:22). Kampos pottery was first found at the Kampos cemetery on Paros, but has now been found at other sites, such as the settlement of Markiani on Amorgos and the Agrilia on Ano Kouphonisi (Marangou et al. 2006; Zapheiropoulou 1984, 2008). Kampos vessel forms, such as the fruitstand and frying pan, appeared on the mainland in late EBA I, although the frying pans were likely made locally and derived from Cycladic types (Coleman 1985:201; Pullen 1995:13, 2008:22; Dousougli 1987).

Early Bronze Age II (2650-2200 BC)

Early Bronze Age II society has been extensively investigated and is the best-known period of the Early Bronze Age. An "international spirit" characterized the period, as sociocultural innovations appeared across the Aegean (Pullen 2008:24; C. Renfrew 1972:34). This period can be broken into two phases, EBA IIA (2650-2500 BC) and EBA IIB (2500-2200 BC) that mostly correspond to differences within the ceramic assemblage and, at least on the mainland, to changes in architecture. EBA IIA is associated with the Korakou culture on the mainland and Keros-Syros in the Cyclades, while EBA IIB with the Kastri/Lefkandi phase.

In EBA IIA on the mainland, settlements were proto-urban in character and were nucleated (Kouka 2008:276). The number of settlements and other sites increased in the Peloponnese, the Attic coast, and Euboea (Kouka 2008:277; C. Renfrew 1972:107). The Attic/Euboean settlements could be described as Helladic, but with Cycladic features (Kouka 2008:277). There is a possible hierarchy of sites, at least in the Argolid, with three to four levels of sites that range in size, material culture present, and distance to one

another (Pullen 2008:27). The use of the plow and traction animals, as evidenced by yoked oxen figurines from Tsoungiza, and expansion of settlements into low hills and uplands reflect changes in agriculture (Pullen 2008:28). It is likely that the corridor house, the architectural hallmark of the next phase (EBA IIB), was developing in this period. Shaw (2007:138) argued that the house at Thebes is likely the earliest datable corridor house. Pullen (2008:29) argued that House A at Tsoungiza is a slightly later precursor to the corridor house. House A was a one-roomed house with thick stone foundations, a corridor which may have housed a set of stairs leading to an upper level, and a covered porch with a post (Pullen 2008:28). Both structures share some elements with the later, fully-developed corridor houses; however, they are far from standard construction in the period. Eutresis had a two-roomed structure with hearths in each room, stone foundations, and likely a mud brick superstructure (Pullen 2008:28). Lithares had a settlement of twenty houses, each with one to four rooms, aligned on a street with shared walls between the units (Tzavella-Evjen 1985). Widespread metallurgy is seen, as well as possible administrative control in the form of sealings (Pullen 2008:30). Communal feasting is suggested at the 'Burnt Room' at Tsoungiza based on specialized drinking sets of small bowls and pouring vessels and paleoethnobotanical evidence for food preparation in its final stages (Wright et al. 1990; Pullen 2008:30). These developments hint at a greater social complexity than the previous period.

The mainland in the EBA IIB period is characterized by complex architecture, fortified sites, and widespread contacts. Monumental architecture was fully realized in the form of corridor houses and fortifications became common, even on small sites (Forsen 2010:61; Pullen 2008:31). The type site is Lerna in the Argolid, a community of

around 50-110 households (Pullen 2008:35). The first fully developed corridor house appeared at Lerna with the structure named the House of Tiles and became a widespread architectural form. It was the first structure to use roof tiles in the Mediterranean, possibly even the world (Pullen 2008:34). Corridor houses are large, two-storied structures with thick walls, monumental entrances, corridors flanking the long sides, and distinct public and private spaces with possibly restricted access (Shaw 1987:59-60). The House of Tiles had over 70 sealings in the externally accessed room, thought to belong to surrounding settlements (Pullen 2008:34-35). Corridor houses have also been found at the sites such as Kolonna on Aegina, Akovitika, and Tiryns (Shaw 2007:141). The House of Tiles was demolished later in the period and buried by a tumulus (Caskey 1960:293). Although the cause is unknown, there was an apparent cultural shift at the end of this period moving into EBA III (Pullen 2008:36). This date does correspond to environmental changes at the time in other areas, which may have been the impetus (Manning 1997; Pullen 2008:36).

In the Cyclades, the archaeological evidence points to a different settlement pattern. In this period, Cycladic culture reached its peak or Golden Age (Doumas 1977:69; Kouka 2008:276). Population increased during EBA IIA and the number of sites grew (Doumas 1977:14). Although a few examples exist, large village sized-settlements were the minority in the Cyclades. Most of these islands were populated by small, isolated hamlets, no larger than a fraction of a hectare, with a handful of families occupying them as in the preceding EBA I phase (Broodbank 2000, 2008:54). EBA IIA houses were rectangular structures with walls made of slab-like stones and clay (Doumas 1977:14). The population of average-sized Cycladic islands has been estimated between

37-225 people (Broodbank 2008: 54). Some scholars have argued that a group of this size was not large enough to sustain itself in marriage and reproduction and therefore depended on interregional social networks and trading relationships for long term survival (Broodbank 2008:54). Others have argued that as few as 50-100 people make up a genetically viable group if social practices, such as the incest taboo, are nonexistent and polygyny or extramarital pregnancy is allowed (MacCluer and Dyke 1976:7). Mixed farming was supplemented with the consumption of grapes and olives (Renfrew 2010:90). Metal objects witness a resurgence from their near absence in EBA I (Broodbank 2008:63; Kouka 2008:278). Gold has not been found on the islands, but lead, copper, silver, and bronze objects are well attested (Barber 1987:135; Broodbank 2008:63). Trading increased overall, as short-range trading intensified and long-range trading was picking up steam (Broodbank 2008:54; Kouka 2008:276). Intense multidirectional relations with the outside world were evidenced by resemblances between finds on the Cycladic islands and the Peloponnese, Troy in Western Anatolia, and Liman Tepe in Southwestern Anatolia, for example (Renfrew 2010:88-89). Sophisticated marble figurines called folded arm figurines were developed from earlier more schematic antecedents in this period in addition to the increase in marble vessels and bone tubes (Barber 1987; Kouka 2008:276 and references therein). Mainland imports to the Cyclades in the form of fine Urfirnis sauceboats and yellow-mottled ware are evident, although usually a small fraction of the pottery assemblage at the smaller sites (Broodbank 2008:64-65). Exports from the Cyclades—sauceboats, marble figurines, bone tubes, liquid storage vessels—formed the currency of prestige goods that were circulated widely (Broodbank 2008:63).

There were a few larger settlements that stood out in this period on the Cyclades: Ayia Irini on Keos, Grotta on Naxos, Kavos and Daskaleio on Keros, and Skarkos on Ios (Broodbank 2008:55). None of these had corridor houses (Broodbank 2008:56). Ayia Irini had well-constructed houses neatly organized (Wilson 1999). Skarkos had two-story houses with communal spaces (Broodbank 2008:55). These were likely village-sized communities with 200-300 people in closely packed households (Broodbank 2008:55; C. Renfrew 1972:251; Whitelaw 1983:332-333, 1991:207-208).

These villages displayed a level of craft specialization, prestige goods, and conspicuous consumption (Broodbank 2008:56).

It was previously thought there was a break in occupation after the Keros-Syros culture, but in fact there is continuity with the Kastri group (C. Renfrew 1972:533; 2010:89). Kastri on Syros is a major settlement in this period. It, like many sites belonging to the Kastri group, was fortified (Renfrew 2010:89). Tin bronze was used more often than arsenic bronze in this period (Nakou 1995). Anatolian forms and fabrics were added to the assemblage, which again reflects more intense contacts with western Anatolia in a wider network than seen earlier in the EBA IIA period (Kouka 2008:278). The nature of these contacts is not entirely clear. The influx of Anatolian shapes could indicate movement of Western Anatolian groups or specialists, but this is not a necessity (Kouka 2008; Rutter 1979:14).

The social structure of EBA II mainland society is still argued upon by scholars. Arguments seem to hinge on how one interprets the corridor houses on the mainland and the larger settlements on the Cyclades. Pullen (2008:34) and Wiencke (1989:508) argued that corridor houses were regional administrative centers belonging to chiefdoms and

inhabited by the elite. Others have argued that corridor houses were community centers used by various groups at various times, but without hierarchical connotations (Nilsson 2004:208-214; Peperaki 2004:226; Weiberg 2007:53). Erkanal (2008:183) argued that they were religious centers, while Felten (1986:26) argued that corridor houses were living quarters for large family groups. Current thought is that the corridor houses were used by multiple groups at different times, a theory which coincides with the possibility that more than one corridor house may have been in use at the same time within a settlement (Maran and Kostoula 2014:151; Weiberg 2007:39). Pullen (2008:34) and Wiencke (1989:508) interpreted the corridor houses to be the seats of small, regional chiefdoms, which dotted the landscape and coexisted, where the elites controlled and redistributed resources. Wienke argued that there was no one dominant settlement; instead, chiefdoms were basically equal and independent from one another, but had a competitive relationship (Wienke 1989:508). There was growth in the use of seals for administrative control and redistribution (Pullen 2008). Regardless of their exact use and role within the community, corridor houses reflect a certain level of social organization that was needed for their construction (Tankosić 2011:51). Couple this sophisticated architecture with the possible use of sealings for administrative control, which Maran and Kostoula (2014:154) argued may have given rise to a penal system, and extensive interactions around the Aegean and a more complex society emerges. Regardless of the nature of EBA II society, this period seems to be the "most complex social and political organization on the mainland until the beginning of the Mycenaean period several centuries later" (Pullen 2008:30).

Whether or not social ranking existed at the larger sites in the Cyclades is still debated as well. Broodbank (2008:56) argued that the larger Cycladic settlements were roughly egalitarian, without ranking as an institutionalized system, hereditary leaders, or overarching administrative control. However, Renfrew (2010:90) suggested that seals from Zas Cave and Skarkos reflected social ranking and administrative control. Regardless, Broodbank (2008:65-66) reasoned that social hierarchies would have derived from maritime exploits on the Cyclades and not ancestral or hereditary linkages. The success of these villages was probably linked to their prominent position within trading networks (Broodbank 2008:64). The Cyclades were poised well to engage in interregional trade, since a significant amount of maritime traffic had to pass by them (Broodbank 2008:47-48). Broodbank (2000, 2008) used proximal point analysis to discuss nodes of movement across the Cycladic islands and showed that the location encouraged the expansion of some settlements into large emporia, such as Ayia Irini on Keos, while it discouraged others.

In terms of pottery, a wider range of shapes and decorations were present in the EBA II period than the previous periods (C. Renfrew 1972). EBA II is characterized by the development of high quality pottery with dark semi-lustrous slip, called 'Urfirnis' pottery, and yellow-mottled ware (Forsen 2010:53; Pullen 2008:26; C. Renfrew 1972:100). Characteristic shapes were thickened or incurving rim bowls and saucers, collared jars, flared rim pithoi sometimes with rope band decoration, cylindrical pyxides, and sauceboats; the latter was possibly connected with communal feasting on mainland and the Cyclades (Barber 1987:92; Broodbank 2008:61; Pullen 1995:20, 32-33; 2008:26; C. Renfrew 1972:100, 112). Sauceboats resemble modern gravy boats with their deep

one-handled bowls and spout, which on some examples is so exaggerated that it causes the vessel to be unstable (Theodorou-Mavromatidi 2007:248). Specialized drinking sets appear, such as in the abovementioned Burnt Room at Tsoungiza (Pullen 2008:30). Although still handmade in this period, pottery was made with finer fabrics and with higher firing temperatures (Broodbank 2008:60). The fruitstand disappears on the mainland as well as certain fabrics known in EBA I (Pullen 2008:26). Impressed decoration and dark painted designs on lighter fabrics appeared, while burnishing, incising, and filling in with white paste continued to be common decorations (Broodbank 2008:61; C. Renfrew 1972:101). Ring bases are common in this period (Pullen 1995:34).

The *end* of Early Cycladic/Early Helladic II is the period also known as the Kastri/Lefkandi I phase (Rutter 1979). Until Rutter's (1979) reanalysis of the ceramics from this time period, these were thought to be two separate pottery groups with different geographical loci, Lefkandi I from Euboea and Attica, and Kastri from the Cycladic islands. Rutter (1979:6) argued that the Lekfandi I and Kastri groups were actually the same pottery assemblage. They were not recognized as the same group initially, because the full suite of characteristic shapes was not found at each site. The sites with the most complete 'package' of Kastri/Lefkandi I shapes are the sites of Manika and Lefkandi, both on Euboea (Wilson 1999:100). The Lefkandi I/Kastri phase includes 'Anatolianizing' shapes, those shapes that show strong similarities with Anatolia: plates and shallow bowls, one-handled tankards, two-handled cups, depas cups, beaked lentoid jugs, and incised *pyxides* (Rutter 1979:6). A rare shape is the wheel made plate, which is found in Central Euboea. Interestingly, the Lefkandi I/Kastri pottery is mostly found in funerary contexts at Manika (Kouka 2008). Most of the shapes are handmade, but the

first use of the pottery wheel is attested (Rutter 1979:8). The Lefkandi I assemblage represents a shift in drinking shapes from the earlier period (Rutter 1979:8-9).

Thus, EBA witnessed social, technological, and material cultural changes, reaching its height in EBA II. As I have tried to show, these changes did not appear spontaneously, but rather were initiated as early as the LN II/FN period. Now I delve further into two areas of scholarship pertinent for this study: diet and burial traditions of the mainland and Cyclades in LN II/FN and EBA.

Diet of the Final Neolithic and Early Bronze Age

The diet of the people who lived in Final Neolithic and Early Bronze Age is not as well known as later periods in Greece, such as the Late Bronze Age (LBA) or Classical periods (Megaloudi 2006). Palaeoethnobotanical remains are scarce from the EBA, especially in contrast to the large amount of evidence from the earlier Neolithic period and the later LBA (Megaloudi 2006). Megaloudi's (2006) diachronic survey of all palaeoethnobotanical remains from the Early Neolithic through Classical periods underscores this point. Of the 79 sites covered in this volume with remains, only 12% date to the Early Bronze Age, while roughly 21% each belongs to the earlier FN and later LBA (Megaloudi 2006: Figure 4.18). Given that the EBA spans nearly 1,000 years, very little data exist with which to reconstruct plant component of the diet. Out of the few sites dating to EBA in Megaloudi's volume, most were in Northern Greece or Crete and therefore, from different cultural and geographic spheres than Euboea.

Sites within the same cultural sphere as Ayia Triada—those from central and southern mainland Greece and Euboea—from which paleoethnobotanical remains have

been recovered are Kephala, Saliagos, Lerna, Eutresis, Tiryns, and Skoteini Cave. Kephala on Kea in the LN II/FN had remains of barley and grass pea (Megaloudi 2006: Table 5.8; Renfrew 1977). Saliagos in the LN II/FN had barley, including the two-rowed variety, emmer and einkorn wheat, and darnel ryegrass (Megaloudi 2006: Table 5.8; Renfrew 1968). Lerna in the EBA had large amounts of barley (including the six-row naked variety), lentils, fava beans, and grapes, with smaller quantities of wheat, pea, bitter vetch, chickling vetch, fig, flax, acorn, and cotton thistle (*Onopordum* cf. *acanthium)* (Hopf 1961, 1962; Megaloudi 2006: Table 5.9). Eutresis had remains of wheat (Megaloudi 2006: Table 5.9; Mylonas 1959). At Tiryns, barley, fava, emmer wheat, lentil, bitter vetch, olive, oats, corn gromwell, darnel ryegrass, crab apple, and little bur-clover were found in minor quantities, and fig and grape in larger quantities (Kroll 1982; Megaloudi 2006: Table 5.9). The only plant remains that have been recovered from Euboea are from funerary contexts, much like Ayia Triada. Skoteini Cave at Tharrounia in central Euboea produced three grains of rye dating to the Late Neolithic, although it has been suggested that they may have been used for fodder (Megaloudi 2006:49; Mangafa 1993). Free-threshing wheat, emmer and einkorn wheat, grass pea, pea, fava, fig, pear, milk vetch, true grass, and green foxtail have been found in the cave dating to the FN (Megaloudi 2006: Table 5.8; Mangafa 1993). Dating to the EBA at Skoteini are fava beans, barley, emmer and einkorn wheat, free-threshing wheat, spelt, chickpea, grass pea, pea, lily, and false cleavers (Megaloudi 2006: Table 5.9; Mangafa 1993). Fava beans were found in the highest concentration out of all remains at the cave, which led the excavators to suggest that it was the principle crop in the area (Mangafa 1993). Ayia Triada's plant remains will be discussed in the latter half of this chapter.

Since there is so little evidence for plant remains on Euboea, the nearby islands, or adjacent mainland, it is useful to discuss diet in broader terms across the Aegean. In general, wheat dominates plant assemblages in LN II/FN. Emmer wheat and einkorn wheat have been recovered from almost every site where plant remains were collected (Megaloudi 2006:75; Valamoti 2007: Table 6.1). Barley is also common with the two-rowed variety being replaced by the six-rowed variety (Hansen 1988:44; Megaloudi 2006:75). Barley may have been the dominant cereal in the Cyclades (Hansen 1988:44). It is the most dependable in climates with low rainfall when compared to wheat; it can also tolerate some salinity and poor soil (Megaloudi 2006:75; Sarpaki 1992:69). In terms of wheat varieties, einkorn and emmer wheat are the most adaptable to poor climate and soil conditions (Sarpaki 1992:60). People living at some sites seemed to prefer one wheat over the other. In Northern Greece, there seems to be a general preference for einkorn wheat (Valamoti 2007:95). Spelt wheat, timopheev wheat, and rye appear in the LN, although rye has only been found at one site (Megaloudi 2006:75). Millet appears at one site in the FN in Northern Greece, in a miniature pot in Olynthus, but does not appear to be cultivated until the Late Bronze Age (Mylonas 1929; Valamoti 2007:99, 2009:26). Lentils, bitter vetch, grass pea, chickpea, and peas are common pulses from assemblages dating to this period, with lentils being most common (Megaloudi 2006:75; Valamoti 2009:27). It is interesting to note that bitter vetch and grass pea need extra processing to remove toxins before consumption (Valamoti 2007:96, 2009:29-30). Fava or broad beans begin to be used in LN II/FN as well (Megaloudi 2006:75). Figs and grapes were possibly cultivated (Megaloudi 2006:75; Valamoti 2009:27). Grapes may have been used to make wine as early as FN, as evidence for grape pressings were found at FN Dikili Tash in

Macedonia (Mangafa et al. 2002). Flax and terebinth nuts were probably used for oil, with flax likely being the most common (Valamoti 2007:89,96; 2009:27). Poppy may have been used for oil, but the evidence is scant (Valamoti 2007:99). Wild plants, such as hazelnuts, pears, purslane, coriander, blackberries, capper, elder and dane wort fruits, almonds, and acorns were gathered (Megaloudi 2006:66,75; Valamoti 2009:28). The olive tree is attested in the pollen record in southern Greece in this period and possibly it was used for its oil or fruits (Bottema and Moody 1987; Gennett 1982; Moody 1987).

Plant remains in the Early Bronze Age are patchy at best (Megaloudi 2006). The evidence we do have points to a continuation of crops used in the LN/FN, with barley, emmer and einkorn wheat, and pulses (Megaloudi 2006:76). In addition, spelt wheat became popular in the Northern mainland (Megaloudi 2006; Valamoti 2007:98). Free-threshing wheats were used in small numbers in EBA (Megaloudi 2006: 76; Valamoti 2009:26). Fava bean consumption increased, but lentils and bitter vetch continue to be the most common pulses overall (Megaloudi 2006:76). Grapes were likely being intensively cultivated in the Early Bronze Age in southern Greece, but the palaeoethnobotanical record indicates they were not domesticated until the Middle or Late Bronze Age (Hansen 1988:48). We have more evidence for grapes being used for wine in the form of grape pressings from EBA Myrtos on Crete and Ayios Kosmas on the mainland, but whether wine was produced from them is still debated (J.M. Renfrew 1971, 1972; Valamoti 2009:33). Wild fruits, nuts, and herbs were collected, such as figs, blackberries, pistachios, acorns, cornelian cherry, elder and dane wort fruits, and coriander (Megaloudi 2006:76; Valamoti 2009:28). The most likely oils used were flax and *Lallemantia*, while it is possible that olive oil was used on Crete (Hamilakis 1996; Megaloudi 2006). Pollen

cores from mainland Greece suggest olive cultivation, but olive pits dating to EBA levels have only been found at Tsoungiza (Hansen 1988: 45; Megaloudi 2006:59 and references therein). There was at least a slight diversification of crops, particularly of legumes, starting in the LN II/FN and continuing into EBA, although the extent to which this was purposeful rather than the result of the natural movement of species is debated (Hansen 1988:44,48; Megaloudi 2006:76; Valamoti 2009:27).

Turning to the meat component of diet, goats, sheep, cattle, and pigs were consumed in both the LN and EBA on the mainland and the islands (Broodbank 2008:53). These animals could also be exploited for secondary products such as wool or dairy, a practice which started in the Late Neolithic (Sherratt 1981). Fish and wild animals would have been a minor component of the diet (Broodbank 2008:53). However, people consumed fish frequently from at least one site, as evidenced by a large quantity of bones from tuna at Saliagos near Antiparos (Evans and Renfrew 1968). It is generally thought that meat was only consumed in large quantities in ceremonial or ritual contexts (Hamilakis and Sherratt 2012:190).

Preparation and consumption methods of foods can be deduced from plant and animal remains and from pottery shapes (Rice 1987; Arnold 1985). Again, we struggle with limited evidence, however, as to what this looked like in EBA II. In general, we do see an increase in the Final Neolithic in the proportion of coarse-ware storage vessels to fine-ware vessels, which suggests possible innovations or shifts in processing and storage of food (Cavanagh 2007:117). This continues into the Early Bronze Age. Cavanagh (2007) has speculated maybe brewing or fermenting was conducted in these coarse-ware vessels, especially the ones with holes around the rim. Analysis of feasting remnants

when present can also speak to foodways, but rarely do we have feasting episodes preserved in the archaeological record in EBA Greece. More often we have production remains, such as the byproducts of dehusking wheat, that are preserved instead of consumption remains.

Burial Practices in the Mainland and Cyclades

Neolithic

Burial practices vary diachronically and regionally between the mainland and Cyclades, as these cultures developed their own unique traditions for burying the deceased. Evidence for burials in the earlier phases of the Neolithic is scarcer. Neolithic society was considered roughly egalitarian, which could have contributed to the lack of lavish burials, but would not explain why so few burials have been found (Cavanagh and Mee 1998:7; Mee 2010:278). Mee (2010:278) suggested that maybe interments were generally not that deep and therefore have been destroyed by modern activity or were more susceptible to erosion or other natural disturbances. Although adults have occasionally been buried intramurally, this burial type was mostly reserved for infants and children (Cavanagh and Mee 1998:7; Mee 2010:277). When human bone is found at NL sites, it is usually from secondary inhumations, although some cremations have been found sporadically in the region (Cavanagh and Mee 1998:5-7). Scattered human bone has been found at Alepotrypa and Franchthi Caves, for example, buried amongst the habitation zones. None of the known NL burials suggest any social distinctions among individuals or families (Cavanagh and Mee 1998:10). Grave offerings, mostly vessels for food consumption, are only found in some of the burials (Cavanagh and Mee 1998:11).

It is not until later in the LN II/FN that people first buried their dead in cemeteries in the larger region (Cavanagh and Mee 1998:10; Doumas 1977:69). As a result, we have a much clearer picture of burial practices in this period than in LN I. On Keos in the Cyclades, a community cemetery at Kephala has been found with a range of individual to multi-person corbelled graves, some with platforms thought to have been used for funerary rituals (Cavanagh and Mee 1998:7-8; Coleman 1977). Children were buried in their own graves or with adults, while infants were buried in jars in separate areas of the cemetery. Also, at the settlement of Tharrounia, Euboea, a cemetery of seven irregularly shaped cist graves were found dating to the LN II/FN (Sampson 1992). Both cemeteries seem to have family tombs (Cavanagh and Mee 1998:11; Coleman 1977:44, 135).

Dating to the end of LN II/FN and continuing into EBA I, the cemetery at Tsepi, Marathon in Attica contained around 70 tombs with both Cycladic and Helladic attributes (Pantelidou-Gofa 2005: 324-327, 338-339; Petrakos 2007:20). Multiple inhumations were found in small cist graves capped with stone slabs and enclosed with neatly laid low wall of stone; these enclosures are unique features of Tsepi (Kapetanios 2010:32). There were symbolic doorways on the south side of the cist graves; they were blocked and too small to be functional, suggesting that the corpses were posited into the grave vertically (Marinatos 1970:154). The graves were organized in clusters that were oriented NE-SW, which could represent families or larger kin groups. Secondary burials were found outside the cist graves, mostly within the enclosure (Pantelidou-Gofa 2005). The back of most of the chambers held a pile of bones and sometimes the skulls were stacked up separately along one of the walls (Mee 2010:278). Grave goods overall were not extensive, but did include ceramic vessels, metal jewelry, and weapons. A depository pit

was found with over 1,000 utilitarian vessels of good quality that were smashed with rocks *in situ* and many of them burned prior to their placement in the pit; Pantelidou-Gofa (2008: 282-283; 285) interpreted this as a pit for burial offerings. Earlier tombs were even reconstructed to conform to the standard pattern once it was established at Tsepi (Pantelidou-Gofa 2005:296, 337, 241).

Early Bronze Age

Unlike the NL, many burials have been found throughout the long EBA period and clear regional differences exist. Focusing on the southern mainland first, very few graves have been found dating to EBA I or EBA III (Cavanagh and Mee 1998:15). However, in the intervening EBA II, the overwhelming trend was to bury the dead in cemeteries of rock-cut chamber tombs, cist graves, and in a few instances, hollowed pits in bedrock with occasional intramural infant burials (Cavanagh and Mee 1998:15; Pullen 1994:126). Cist graves are subterranean, slab-lined rectangular or trapezoidal graves, while built tombs are generally a bit larger, rectangular in shape, and built with rubble walls instead of slabs. Multiple burials per grave were common, usually representing families or corporate groups (Pullen 1994:126). The individual is usually buried in a flexed position, regardless of the size of the tomb (Pullen 1994:126). A stone 'pillow' slab supported the head in many graves (Canavagh and Mee 1998:20). When a new person was buried, earlier burials were pushed to the side to make room. Cemeteries are very visible on the landscape in distinct areas away from the settlement on the mainland, even if only a few hundred meters away (Pullen 1994:126). It is not clear if all of the dead were formally buried, because not all of the settlements to which the cemeteries

belong have been located and therefore, it is impossible to gauge (Pullen 1994: 126). The majority of cemeteries that have been excavated are from the eastern mainland, in areas of Attica, Boetia, Corinthia, Argolid, as well as on Euboea (Canavagh and Mee 1998:15-21; Mee 2010; Pullen 1994).

Ayios Kosmas in Attica, and Manika on Euboea are well-known Early Helladic cemeteries. There is considerable variation among graves, not only between cemeteries, but also within them. Tombs at Ayios Kosmas were cist or built graves with symbolic entrances and passages (Canavagh and Mee 1998:16; Mylonas 1959). Two separate groups of graves were found, one in the northern part of the cemetery and another in the southern part. Cist graves were on the coast, while the built graves were placed more inland (Mylonas 1959:115). These graves were likely intended for family groups; they were mostly primary inhumations for up to 16 individuals, ranging from children to adults, with skeletons pushed to the side when a new skeleton was interred (Canavagh and Mee 1998:19; Mylonas 1959:117; Pullen 1994:123). Interestingly, many of the grave goods were placed on the ground atop a layer of intentionally arranged stones (Mylonas 1959; Pullen 1994:123). Grave goods consisted of both Cycladic and Helladic pottery vessels, stone bowls and figurines, obsidian, and shells (Mylonas 1959:68-71). Pullen argued for differential treatment of the dead based on status, citing three human burials outside of the family graves (Pullen 1994:123-125). However, Mylonas (1959:118) argued for a multi-stage burial ritual, citing especially one articulated and extended skeleton.

Manika tombs, which number over 170, were mostly rock-cut chamber tombs with vertical or sloping shafts called *dromoi* (Canavagh and Mee 1998:17). At Manika,

there is evidence for manipulation and cutting of the human remains to get them into the flexed position, which was performed on all sexes and ages from both rich and poor graves (Fountoulakis 1987; Sampson 1985). Some tombs held multiple individuals, while others only held one individual (Sampson 1985:226). Six distinct clusters of tombs can be delineated by their layout and structural differences (Doumas 1977:67). Sampson (1985:222) doubts that these are family tombs because they held mostly juveniles and females. Some tombs share the same dromos (Canavagh and Mee 1998:17). Grave goods consisted of pottery, stone vessels and figurines, metal weapons and tools, obsidian blades, and bone tubes and were usually placed by the skull (Sampson 1988:49-53). The quality and quantity of these grave goods vary from tomb to tomb and may reflect differential status (Sampson 1985:27). Some bones might have been removed from the grave altogether during the subsequent burials. Most of the graves are considered 'poor' by Sampson with regard to the grave goods.

It has been suggested that secondary burial rites accompanied mainland EBA II burials, at least in Attica. Mylonas (1959:118) found a fully extended skeleton in the Ayios Kosmas cemetery. This may be evidence for prosthesis or laying out of the body for the flesh to decompose. Once completed, the bones would have been collected and deposited in the cist or built tomb (Mylonas 1959:118-119). Mylonas (1959:118) argued that these findings represented a two-stage mortuary ritual. Also, at Lefkas, cremations were buried in pithoi with burned grave goods, presumably from the funeral pyre, capped with a stone slab, encircled with a stone platform, and then covered over with a circular tumulus (Mee 2010:280).

Cycladic burials were also concentrated in cemeteries like on the mainland. However, there was far less diversity in tomb types in EBA Cyclades in comparison to the southern mainland (Barber 1987; Doumas 1977). Cemeteries were usually built close to the settlements throughout the EBA and often were on the coast (Doumas 1977:29). It is likely that all inhabitants of the settlements were formally buried instead of just a select group (Broodbank 2000:170). Cist graves were so commonplace in the cemeteries of the Cyclades that connections or even Cycladic colonies have been suggested at other sites, such as Ayios Kosmas, based on the presence of this tomb type (Barber 1987:136; Doumas 1977; Mylonas 1959). The only exception to the use of the cist grave is with the corbelled graves of Syros in EBA II (Doumas 1977:47). Corbelled tombs are built in a pit with dry stacked stones that have a corbelled roof and a symbolic doorway; they vary considerably in plan (Doumas 1977:49). There is at least one cremation burial of an adult known from this period in the Cyclades from Dhaskalio on Keros (Moutafi 2013). The cist graves tend to be less than 1.2 m in length, vary in width, and are rather shallowly buried (Doumas 1977:37; Tsountas 1898:143). Among the cist tombs, Doumas outlined seven types, some of which link to chronological periods (Doumas 1977:41-47). EBA I only has Type A cist graves, while EBA II graves fall into a variety of the cist types, which may correspond to local idiosyncrasies (Doumas 1977:53). Cycladic burials were used mostly for single interments in EBA I; we see an increase in multiple burials per grave in EBA II at every site except at Syros (Barber 1987:77). When another burial was added, the original burial was swept aside, but the skull was left in place (Mee 2010:278). In EBA I-II, a paved floor and a stone pillow were added to the graves, a practice which became commonplace in EBA II (Doumas 1977:52). The use of multi-storied cist graves

began in the late EBA I to EBA II period (Barber 1977:80). The lower 'story' was usually turned into an ossuary, while the new burial was laid on the top story; the second or third stories were added when the next person was interred (Barber 1977:81). Bodies were usually buried in a strongly contracted position in EBA I-II, although the space did not require this placement (Mee 2010:278). This indicates a possible binding of the body (Barber 1987:80; Doumas 1977:54).

The orientation of graves within Cycladic cemeteries did not follow any strict pattern, as the layout of the land dictated how the graves were orientated (Doumas 1977:35). This makes sense given the often rocky, uneven landscape of the Cycladic islands. Graves were often placed on sloping ground with evidence for retaining walls nearby to prevent erosion (Doumas 1977:30). Graves were generally clustered into small groups belonging to either families or extended families (Doumas 1977:31). An example is Chalandriani, where graves were grouped in two main sections with large family clusters (Doumas 1977:32). Most cemeteries contained between 15-20 graves, suggesting small burying groups (Doumas 1977:31; Mee 2010:278). Doumas (1977:31) noted that only eight cemeteries have 50 burials or more, but these seem to represent longer habitations rather than larger burying groups. One notable exception to this pattern is Chalandriani cemetery, which had well over 600 burials (Tsountas 1899:78-79). Social differences in cemetery layout are suggested at Ayioi Anargyroi on Naxos, which is attributed to the transitional EBA I-II Kampos period (Doumas 1977: 34). Well-constructed larger cist graves with richer grave goods were spaced out evenly in the north part of the cemetery, while small, poorly constructed graves were crammed up against the retaining wall with poorer grave goods (Doumas 1977:34). Stone platforms of unknown

function have been found at a few cemeteries. They were likely present at other Cycladic cemeteries as well, but were ignored when excavated; unfortunately, the layout of the cemetery was rarely studied in the earlier excavations (Doumas 1977:36). Platforms were built either one per grave or one per cemetery (Doumas 1977:70). The best example is a 40 m long by 3-4 m wide platform at Ayioi Anargyroi at the southeast part of the cemetery (Doumas 1977:36). Platforms with associated hearths suggests that funerary rituals took place, possibly with food and drink consumption (Doumas 1977).

Many of the cist graves in the Cyclades had no grave goods (Mee 2010:279). No marble figurines, obsidian, or metal objects have been found in the earliest part of EBA I (Barber 1987:81). When grave goods do begin to appear, they usually consist of fine-ware pottery, followed by occasional obsidian blades, marble vessels, and figurines, beads, bronze sewing and grooming tools, and boat models (Doumas 1977:60, 62; Mee 2010:288). Few rich graves have been found at all (Doumas 1977:58). It was more common to have relatively few goods/offerings, which were usually placed in front of the face and were usually items used in everyday life as evidenced by wear marks or repairs (Doumas 1977:62-63). Tombs with multiple burials usually are the poorest in terms of grave goods (Doumas 1977:60). The lack of grave goods should be viewed with caution, however, because of the extensive looting to which Cycladic burials have been subjected in the pursuit of marble figurines. Furthermore, the fact that some of the grave goods could have been perishable items, such as cloth or food, should be taken into consideration (Mee 2010:279).

Southern Euboea

The previous discussion has laid the foundation for our understanding of Ayia Triada Cave. The Ayia Triada excavation is part of the larger archaeological focus on the Southern Euboea region. Euboea is the second largest island in Greece and is separated from the mainland by the Euboean Gulf (Figure 2.1). It has numerous water sources and bays (Talalay et al. 2005:31). Until relatively recently, the region was thought to be uninhabited throughout prehistory (Talalay et al. 2005:14). Extensive surveys conducted for the last 30+ years by the Southeastern Euboea Exploration Project (SEEP), which was codirected by Donald Keller and the late Malcolm Wallace, have transformed our understanding of the region and characterized the prehistoric occupation (Cullen et al. 2011, 2013). Four areas have been surveyed on the southern tip of Euboea: the Paximadi Peninsula, Kampos Plain, Borous-Kastri Peninsula, and Katsaronio Plain (Cullen et al. 2011, 2013; Tankosić and Storli 2013). Prior to these surveys and excavations, some small-scale surface surveys had been undertaken that spurred interest in the island (Talalay et al. 2005:21 and references therein). Most of the prehistoric find-spots have been dated to the FN-EBA, when artifacts have been in good enough condition to date (Cullen et al. 2011, 2013; Talalay et al. 2005). Southern Euboea has produced over 60 sites and find-spots from the prehistoric periods (Cullen et al. 2013; Žarko Tankosić, personal communication 2017; Tankosić and Storli 2013). Most of the spots were occupied for only one period, probably short term, and were relatively small (Cullen et al. 2013:48, 88-89; Talalay et al. 2005:24, 31). Survey data have revealed some general settlement patterns. During LN II/FN, sites were selected at higher elevations on ridge tops and slopes near springs, while in the EBA, sites were focused on the coasts (Cullen

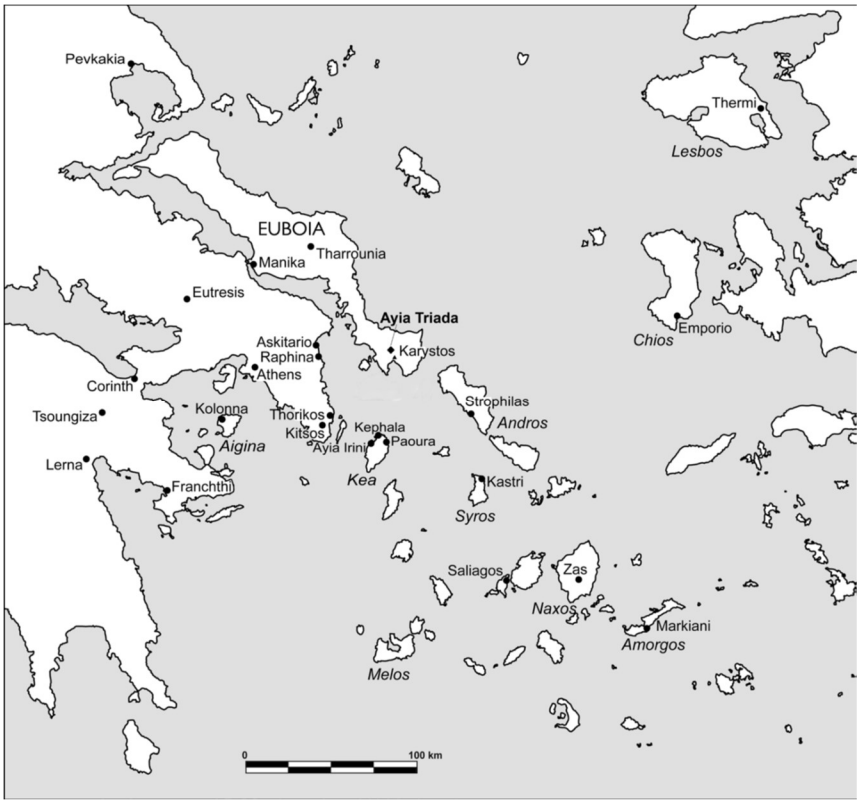

Figure 2.1 Map of Ayia Triada located within the wider Aegean region. (Mavridis and Tankosić 2016a, Figure 1 [adapted from Cullen et al. 2013, Figure 1])

et al. 2005:25). We do not know where people settled in LN I, as this period is only represented at Ayia Triada Cave. Whether Karystia was occupied continuously from FN to the EBA is unclear as well (Cullen et al. 2013:87).

The Paximadi Peninsula is the western half of the Karystian bay. It represents a marginal landscape with its dry and rugged hills (Cullen et al. 2013:90). Survey of this area revealed 11 find-spots and nine sites dating to FN-EBA (Cullen et al 2013:48; Cullen et al. 2011:30; Talalay et al. 2005:23). Most can be interpreted as seasonal or short-term occupation (Cullen et al. 2013:90). Many of the sites would have been visible from one another and would have offered, as they still do today, sweeping views of the nearby mainland of Attica, nearby islands of Kea and Andros, and the surrounding areas of Karystia (Cullen et al. 2013:88). Four of these sites, all located on the northern part of the peninsula, have architectural remains and can be considered more substantial; these will be discussed further below (Cullen et al. 2013:48; Cullen et al. 2011:30; Talalay et al. 2005:23). In addition, two other sites in the Paximadi Peninsula were noteworthy: Cape Mnima and Ayia Paraskevi (Talalay et al. 2005:28-29). Obsidian was found in substantial quantities at Cape Mnima in various stages of the production sequence. It would have been an ideal landing point from the south, possibly for vessels bringing Melian obsidian (Talalay et al. 2005:28). Ayia Paraskevi may have been a fishing camp dating to FN-EBA (Talalay et al. 2005:28). Talc-ware was found at this site, which was likely a Cycladic import, in addition to obsidian tools (Cullen et al. 2013:49). The density of sites across the Paximadi is generally low, especially considering the hundreds of years of occupation that the sites represent (Cullen et al. 2013:88).

The Bouros-Kastri Peninsula flanks the Karystian Bay on its eastern side opposite the Paximadi Peninsula (Cullen et al. 2011:34). It was extensively surveyed in 1989, 1990, and 1993, during which 11 FN-EBA find-spots were found scattered on both sides of the peninsula (Cullen et al. 2011:34, 2013:98-99). Scant architectural remains were found at four sites: Ayioi, Kalamos, Bouletza, and Spilia. They could not be more precisely dated than FN-EBA (Cullen et al. 2011:35). Across the Bouros-Kastri Peninsula, sites are small and dispersed along the coasts or near springs, while some are located in the uplands (Cullen et al. 2011:35; 2013:99). A possible cist grave was found at the EBA II site of Kalamos (Cullen et al. 2013:99). Pottery found across the sites includes saucers, fruitstands, and pithoi decorated with pattern-burnishing, taenia bands, and *Kerbschnitt* punctuation (Cullen et al. 2011:34-35).

The Kampos survey was undertaken in 2006 through 2008; I participated in the 2007 survey. The Kampos Plain is a large tract of agriculturally productive land, likely one of the largest in the region (Cullen et al. 2011:36). Cullen and authors (2011:36) suggested that the plain may have been "devoted primarily to producing foodstuffs that could be locally consumed or exchanged," which could have been related to the obsidian trade. A total of 14 find-spots, mostly obsidian scatters, dating to FN-EBA were found; no new architectural remains were identified (Cullen et al 2011:35). Pottery was rare and too worn to date with any better resolution (Cullen et al 2011:35). Two important find-spots, 07S28 and 07N35, are worthy of further mention. The former produced 300 obsidian flakes in 50 m x 100 m area on the western part of the Kampos Plain and was probably a tool production site (Cullen et al 2011:36; 2013:100). The latter appears to be a specialized obsidian production site where tools were produced or modified as

evidenced by the mostly pressure-flaked tools (Cullen et al 2011:36; 2013:100). Roughly 6,582 lithic fragments were found on the surface alone in an area thought to be larger than the 100 m x 150 m area originally cited (Cullen et al. 2011:35-36; Tankosić 2011:207; Žarko Tankosić, personal communication 2017). One substantial EBA II site, Ayios Georgios, had been identified on the Kampos prior to the SEEP survey and is discussed below.

More recently, the Katsaronio Plain Survey was conducted with the Norwegian Archaeological Survey in the Karystia (NASK) between 2012 and 2015 (Tankosić and Storli 2013). The Katsaronio Plain, located northeast of the modern town of Karystos, is an agriculturally productive area as well. Study of the finds is ongoing, but preliminary results have been reported. A total of 22 find-spots were found in the first season, at least two of which date to the LN II/FN (Tankosić and Storli 2013). A concentration of possible cist graves was located, but no finds or bones were found and thus, could not be dated (Tankosić and Storli 2013).

Five sites with significant architectural remains have been discovered through excavation or surface survey. Excavations in the region are scarce in Southern Euboea and are mostly limited to rescue excavations in response to or in advance of modern construction projects. Two sites, Plakari and Kazara, are dated to the LN II/FN, two to the EBA, Ayia Pelagia and Ayios Georgios, and another with both LN II/FN and EBA occupations, Akri Rozos. All of these sites are located on the Paximadi Peninsula, except Ayios Georgios on the Kampos Plain (Cullen et al. 2011:31-33; 36). Plakari, Kazara, and Ayia Pelagia are located on the eastern coast of the Paximadi Peninsula and Akri Rozos is strategically located on the western coast of the peninsula.

Plakari was excavated initially in 1979 in a brief salvage excavation when the area was being developed for residential purposes (Keller 1985). The site is located on a slope west of Karystos, near two springs (Cullen et al. 2013:21; Keller 1982:47, 1985:168). Two wall segments, two floors, a destruction pit with charcoal, sheep/goat and cattle bone, and a fragmented human occipital bone were found likely dating to the LN II/FN (Cullen et al. 2013:24, 39; Keller 1982:48, 1985:169; Talalay et al. 2005:26). More recent excavations since 2010 have focused on the post-prehistoric occupation of the site, i.e. the Geometric period sanctuary (Crielaard et al. 2012). The Neolithic remains were disturbed by the later sanctuary, farming activities, and modern road construction (Cullen et al. 2013:22). Pottery with parallels to other LN II/FN sites, such as in Attica and on Kea (Kephala), obsidian debitage and flakes, non-local honey flint possibly from Western Greece, and a lump of copper and iron ore were found (Cullen et al. 2013:26, 42; Keller 1982:48, 1985:169). The obsidian was knapped on site from raw nodules that were probably procured from Melos (Cullen et al. 2011:32; Talalay et al. 2005:26). Pottery includes cheese pots, pattern-burnished ware, red-slipped and burnished wares, coarse-ware vessels, a serrated rim, and the unusual 'oatmeal' ware (Keller 1982; Cullen et al. 2013:24). Fine wares seem to be concentrated in area I, while the coarse-wares seem to be concentrated in area II; these suggest that different activities were undertaken in the two areas (Cullen et al. 2013:42). The site is estimated to be a half hectare and would have sustained no more than a few families (Cullen et al. 2011: 31, 2013:42). Plakari was likely a year-round, permanent settlement (Cullen et al. 2013:42).

Kazara is thought to be a LN II/FN seasonal outpost contemporary with Plakari and was identified through surface survey. It is at a higher, more remote elevation about

800 meters west of Plakari (Talalay et al. 2005:27-28). Stone rubble walls and daub fragments were found in association with LN II/FN coarse-ware bowls and jars, and obsidian tools (Keller 1985:100-101, 168; Talalay et al. 2005:27-28). The pottery seems to be utilitarian in nature (Cullen et al. 2013: 50). Again, local knapping and core reduction was suggested by the obsidian finds (Cullen et al. 2011:27). It is thought to be only a seasonal site, because of its precarious location. It would have been ideal as a lookout point for defensive reasons or for herding (Talalay et al. 2005: 28).

Akri Rozos is another substantial site, identified and estimated to be 1.5 hectares in size by survey data and likely occupied year-round (Cullen et al. 2011:33; 2013:58). It has both LN II/FN and EBA components and likely was inhabited in the transition between these periods (Talalay et al. 2005:29). It is on the western coast of the island facing the mainland and opposite the two previous sites discussed. It lies at a strategic promontory along the maritime route from south to north (Cullen et al. 2011:33). It has possible fortification walls that follow the ridge with drains, towers, and bastions, and a wall to the north that would have prevented access from the sea (Cullen et al. 2011:33; Talalay et al. 2005:29). Comparable walls dating to the LN II/FN were found on Andros and on later EBA II sites (Cullen et al. 2011:33; Televantou 2008). The fortification walls could also have been a social display of power in this spot on the landscape visible from the sea (Cullen et al. 2011:34). The pottery assemblage includes local and imported collared jars, footed bowls, deep open vessels, and large closed-mouth containers; other finds include a large amount of obsidian assumed to have been prepared on site from raw nodules, ground stone tools, bronze, copper ore, and clay spindle whorls (Cullen et al. 2013:94; Cullen et al. 2011:34; Talalay et al. 2005:29). Metallurgy was possibly

undertaken at this site (Cullen et al. 2013:58). The coastal bluff surrounding Akri Rozos could have supported a population of 300-450 (Cullen et al. 2013:90).

Ayia Pelagia dates to the FN-EBA II period and was first surveyed by Keller (1985). This site is on the inner eastern coast of the Paximadi Peninsula, south of Plakari (Cullen et al 2005:30). It was likely a substantial settlement, estimated at 1.4 ha in size (Cullen et al. 2013:51). Architectural remains were visible on the surface in 2000, which prompted a salvage excavation by the Greek Archaeological Service (Cullen et al. 2011:32). Sections of six walls were uncovered (Cullen at al. 2013:52). They belong to two buildings approximately 35 m apart with narrow double-faced walls, a few of which are thick enough to have supported the weight of a second story, and a narrow corridor on the long side (Cullen et al. 2011: 32-33, 2013:52). It has been suggested that these might be smaller versions of the corridor houses (Cullen et al. 2011:33, 2013:52). A single cist grave was found at the site that likely contained multiple burials, which have not survived (Cullen et al. 2005:30, 2013:96). It is similar in size and building construction to other EBA II cist graves on the mainland and the Cyclades and could have belonged to a larger cemetery that has not yet been located (Cullen et al. 2013:54). Although FN-EBA I pottery was found at the site, most of the pottery dates to EBA II (Cullen at al. 2013:51-52). Fine EBA II pottery including Urfirnis sauceboats, footed bowls, taenia-banded rims and one possible yellow-mottled ware were found in association with the site. Spindle whorls and obsidian were also found (Cullen et al. 2011:33; Talalay et al. 2005:30). The pottery suggests it was a part of the cultural group of the wider Cyclades, although it may include imported wares from the mainland (Talalay et al. 2005:30).

Very near to the obsidian find-spot 07N35 on the Kampos Plain was Ayios Georgios. It was known from early surveys, but was later excavated by the Ephorate, because an electrical station was being constructed (Cullen et al. 2013: 99; Keller 1985; Sackett et al. 1966; Sapouna-Sakellaraki 1992). It is the only major settlement with architecture known on the Kampos Plain and dates to the EBA II period (Cullen et al. 2011:36; Cullen et al. 2013:100). It is extensive in comparison to most of the other sites in Karystia, at 1.5-2 ha in size (Cullen et al. 2013:99). The architectural remains include the foundations of multiroom, rectangular structures that were substantial enough to have held a second story and curving walls (Sapouna-Sakellaraki 1992:178). The pottery was incised, impressed, stamped, or Urfirnis; main shapes were bowls, saucers, jars, sauceboats, and pithoi (Blackman et al. 1997-1998:64; Tankosić 2011:120-121, 125). Copious obsidian was found (Cullen et al. 2013:99). Cullen and coauthors (2011:36) argue that it was likely contemporary with the two obsidian find-spots nearby, although the relationship is unclear.

Talalay and coauthors (2005:31) suggested that Southern Euboea followed a different trajectory than the rest of Euboea, being settled only in the Late Neolithic whereas central and northern Euboea were both settled in the Early Neolithic. The settlement in the LN II/FN corresponds to the general trend in this period of the settling of the Cycladic islands (Broodbank 2000; Talalay et al. 2005:31). Southern Euboea is isolated from the rest of the island by mountains and is surrounded on all sides by water and therefore, "functioned very much as an island" (Talalay et al. 2005:32). The central and northern parts of the island are geographically closer to the mainland than the southern tip, which sharply juts away from the mainland (Talalay et al. 2005:32).

Ayia Triada Excavations

Ayia Triada Cave is located below Mt. Ochi, north of the modern coastal town of Karystos and sits 50 m above the church from which the cave derives its name, meaning the Holy Trinity (Mavridis and Tankosić 2016a:210). The cave is relatively hidden from view below, but from the inside has an excellent panorama of the sea and the Bay of Karystos (Mavridis and Tankosić 2016a:211). The marble and schist cave has a narrow entrance corridor approximately 70 m long with a maximum height of 8 m that ends in a crevasse (Figure 2.2). A side chamber called the East Chamber branches off the west side of the corridor, clearly a misnomer (Mavridis and Tankosić 2009a:55; 2016a:211-212). Ayia Triada was excavated between 2007 and 2010 under the direction of Fanis Mavridis and Žarko Tankosić in association with SEEP and the Ephorate for Palaeoanthroplogy and Speleology of Southern Greece (Cullen et al. 2011:37; Mavridis and Tankosić 2016a:207). It was the first targeted excavation of a prehistoric site in southern Euboea (Mavridis and Tankosić 2016a:239). Prior to the 2007-2010 excavations, Ayia Triada Cave was independently visited by Sampson (1981) and Keller (1985). Although both found NL remains, neither noted any EBA remains in the cave (Mavridis and Tankosić 2016a:215).

A total of 12 trenches were excavated during the four seasons of the project. The layout of the trenches was defined by the cave's geography (Figure 2.3). In the accessible parts of the cave, 11 trenches were opened: one immediately inside the entrance of the cave (Trench 6), five at the end of the corridor (Trenches 1, 2, 3, 5, 7), and five in the East Chamber (Trenches 4, 8, 9, 11, 12). Another trench was excavated outside the cave

Figure 2.2 Entrance to Ayia Triada Cave. (Mavridis and Tankosić 2016a, Figure 4; Photo by Ž. Tankosić)

Figure 2.3 Site plan with trenches excavated inside Ayia Triada Cave. (Mavridis and Tankosić 2016a, Figure 7; Drawing by T. Chatzitheodorou)

to the south of the entrance in a rock shelter (Trench 10), but no prehistoric material was found (Mavridis and Tankosić 2016a:216). The trenches were generally 1 m x 1 m in size and were excavated to bedrock, except in Trenches 8, 9, 11, and 12 (Mavridis and Tankosić 2016a:216). Layers were distinguished according to soil differences, except when so thick to warrant subdivision. Some excavated soil, particularly from layers associated with EBA burials, was floated to recover small bones and seeds. Locations were recorded by depth and horizontal position for all human bone, altered bone, stone, metal, and other special finds (Mavridis and Tankosić 2016a:216-217). The trenches at the end of the corridor contained LN I and II/FN material. The East Chamber is divided into two sections (Mavridis and Tankosić 2016a:212). The section deepest into the East Chamber, accessed only by crawling, is the location of Trenches 4, 8, 9, 11, and 12 and subsequently all EBA occupation levels (Mavridis and Tankosić 2016a:212, 217). This section is roughly 6 m x 4 m with a high ceiling that almost forms a pitched roof (Mavridis and Tankosić 2016a:212). Trench 12 was at the edge of the stalagmite restricted corridor that separates the two sections of the East Chamber. These trenches covered roughly 70% of the area in the chamber (Mavridis and Tankosić 2016a:221). The first section of the East Chamber was not excavated.

The East Chamber was covered with a layer of mostly sterile silty sand (Layers 1 and 2) atop a thin layer of stalagmite crust, no more than 0.5 cm thick (Mavridis and Tankosić 2016a:212, 217). This crust was formed by mineral deposition from standing water (Mavridis and Tankosić 2016a:212). In some areas of the chamber, another layer of sand, Layer 3, lies below the crust. This disturbance layer had Roman lamp fragments in it (Mavridis and Tankosić 2016a:218-220). Layers 4, 5a and 5b are those associated with

EBA, with the bulk of the cultural material coming from Layer 4 (Mavridis and Tankosić 2016a:220). Layer 5 was split into a and b, because the soil composition and color were different. The interface between 5a and b represents the edge of the area associated with the human burials: 5b was the area associated with the burials, and 5a, the space outside it (Mavridis and Tankosić 2016a:221). Layer 5b was a dark, dense layer that turned out to be composed almost entirely of carbonized organic remains in quantities rarely seen at prehistoric Greek sites (Mavridis and Tankosić 2016a:223 and note 48). A large number of carbonized figs, likely *Ficus carica*, pulses, and cereals were recovered in excellent preservation and are awaiting full analysis. It was determined that many of the figs were placed in the cave as dried figs, not as fresh fruit (Mavridis and Tankosić 2016a:223). Sheep/goat bones were also found in the EBA levels. Oxidized sand overlays Layer 5b in areas. Layers 5a and 6 were sandy layers with little cultural material. The rare material that was found in layer 6 dates to LN II/FN (Mavridis and Tankosić 2009a, 2016a:224; Mavridis et al. 2010). There is a gap in the archaeological record between LN II/FN and EBA II in the cave, which suggests a hiatus between the periods and is reflective of broader trends in Southern Euboea (Cullen et al. 2013; Mavridis and Tankosić 2016a:224).

The material culture dating to EBA in the cave includes pottery, metal implements, and at least two incised bone tubes (Mavridis and Tankosić 2016a:225-230). A total of 106.5 kg of pottery was recovered from layers 1-5b, mostly from the EBA, and 161 kg from the LN layers. Most of the finds in Layers 3, 4, and 5b are pottery (Mavridis and Tankosić 2016a: Table 1). Dominant pottery shapes are incurving rim bowls and large storage jars with everted lips and squat necks, both of which are typical EBA II

types in this region. Sauceboats, pyxides, and footed cups complete the pottery assemblage, but are present in smaller numbers than the storage jars and bowls (Mavridis and Tankosić 2016a:225-226). These fine wares are also characteristic of the time period. Most of the pottery appears to be local upon initial inspection, although its provenience cannot be determined without petrographic analyses (Mavridis and Tankosić 2016a:227). The clay in the larger region of Attica, Euboea, and Kea fires to the same yellowish red to reddish brown in color (Mavridis and Tankosić 2016a:227). Petrographic analyses are underway to confirm the Karystian origin of the clay (Mavridis and Tankosić 2016a:227, note 76).

A few interesting pieces are worth mentioning here, all of which are painted pottery: a sauceboat, two pyxis lids, a globular pyxis, and a spouted jug (Mavridis and Tankosić 2016a:227). The sauceboat has a yellow buff fabric with painted curving and straight lines. It is very similar to the yellow buff fine-painted ware from Ayia Irini on the neighboring island of Kea, thought to be imported from the Cyclades (Mavridis and Tankosić 2016a:227; Wilson 1999:78). The pyxis lids are painted with sets of parallel lines that intersect one another and form a large cross. The spouted jug has parallel lines that form triangles repeated around the circumference of the jug. The globular pyxis, of which only the top part survives, has a painted linear design. Interestingly, the jug, pyxis lids, and pyxis are all made with similar lighter-colored fabric and have direct parallels from Chalandriani cemetery on Syros (Mavridis and Tankosić 2016a:227; Rambach 2000). They are all thought to be imported, because local clays do not fire this pale of a color (Mavridis and Tankosić 2016a:229).

Other small finds include tweezers, a dagger, and decorated bone tubes (Mavridis and Tankosić 2016a:229). The tweezers are made of bronze and are similar to Branigan's type IIIa (Branigan 1974; Mavridis and Tankosić 2016a:230). The Branigan type II dagger is also made of bronze. It is 7 cm long and has part of an attachment for a hilt. Both the tweezers and dagger are dated to the EBA II period (Mavridis and Tankosić 2016a:230). Two bone tubes were found dating to EBA II; these implements are polished, hollowed out fragments of bone, no longer than 15 cm, that are decorated with incised designs. Bone tubes are common in EBA II across the Aegean, found most frequently in island cemeteries, but their function is unknown (Saliari and Draganits 2013:179). Interestingly, no folded arm figurines or "frying pans", both traditional Cycladic grave goods, were found in the cave (Mavridis and Tankosić 2016a:232).

Disarticulated human bones ranging from juvenile to adult were found mostly in Layer 4, with smaller amounts in Layers 3 and 5b (Mavridis and Tankosić 2016a:220). A total of 213 human bone fragments were recovered, belonging to a minimum number of seven individuals, or so thought initially (Mavridis and Tankosić 2016a:220). This number has now been revised to nine individuals (Žarko Tankosić, personal communication 2017). Two skull fragments were found (Mavridis and Tankosić 2016a:221). Human and animal bones in Layer 4 were found at consistently similar depths, which could indicate that little time passed between each burial (Mavridis and Tankosić 2016a:224). Radiocarbon samples taken from a fig and seeds in Layer 5b and human bone in Layer 3 suggest a date of EBA IIA (Mavridis and Tankosić 2016a: 231, Table 2). The Layer 5b samples date between 2890/2870 and 2635/2575 B.C.; the human bone in Layer 3 dates between 2865 and 2492 B.C. (Mavridis and Tankosić 2016a:230).

Strontium isotope and stable oxygen isotope analyses in addition to DNA analyses are being conducted to investigate geographic origins and kinship of the buried individuals and population mobility (Mavridis and Tankosić 2016a:232, 238).

Mavridis and Tankosić suggest that some sort of ritual occurred prior to burial, possibly a feasting event. Plant and animal foodstuffs were burned and then spread across the floor of the East Chamber while they were still smoldering (Mavridis and Tankosić 2016a:223). Sand was then spread over the burnt organic remains and then the human remains were laid on top. It appears that fine ware and the other non-pottery grave goods were intentionally placed below the human remains on top of the burnt organics, while coarser pottery was placed around the remains (Mavridis and Tankosić 2016a:233). Many pots appear to have been intentionally smashed *in situ*. It appears that the skeletons were left exposed on the cave floor, as evidenced by the formation of stalagmite crust on some bones, although the authors admit that this could be due to later disturbances (Mavridis and Tankosić 2016a:220). A few horizontally lain schist slabs were found either brought in from another part of the cave or from outside; these may have been used to lay on top the burials (Mavridis and Tankosić 2016a:233). A small pit of ovicaprid bones was discovered close to the human remains, while several mandibles are found mixed with the human remains themselves. Mavridis and Tankosić (2016a:239) suggested that the Ayia Triada burials could possibly represent an intermediary phase between death and burial where for practical reasons the body is left for the flesh to fully decompose or for enough resources to be gathered for a feast or for ideological reasons that are difficult to describe further, the body is left exposed (Mavridis and Tankosić 2016a:238). Regardless if rituals were performed or the burials represent an intermediate stage, the combination of

exposed skeletal remains in a cave over a burnt plant and animal layer is not common in this period (Mavridis and Tankosić 2016a:232).

It is unclear how to fully interpret and explain the Ayia Triada case. The bones themselves further complicate the matter. It cannot yet be determined if they represent primary or secondary burials (Mavridis and Tankosić 2016a:237). The skeletons are disarticulated and incomplete; the bones usually lost in the process of reburial elsewhere are present, while the bones not usually lost are missing (Mavridis and Tankosić 2016a:234-235). No pits were found into which the bones were slotted to be buried in the cave and no cemeteries in the vicinity have been found (Mavridis and Tankosić 2016a:232). The only other human remains in the vicinity of Karystia are from Nea Strya and are likely secondary burials with more Cycladic connections than Ayia Triada as evidenced by grave goods (Kosma 2010; Mavridis and Tankosić 2016a:232). Considering that the settlements in Southern Euboea may have been the home to as many as several hundred to a thousand people in EBA and yet only nine individuals were buried here suggests differential treatment of the dead based on some form of social distinction (Mavridis and Tankosić 2016a:237; Tankosić 2011:194). Where the inhabitants of these communities were buried, as well as why these individuals were treated differently than the rest in death remain unanswered questions.

Summary

The inhabitants of Southern Euboea who participated in the burials in Ayia Triada Cave resided and interacted in a fractured cultural landscape situated between mainland Greece and the Cycladic islands to the south. Ayia Triada Cave has material culture and

burial practices that reflect this interaction. Foodways and feasting, especially in burial contexts, are poorly understood in EBA II. Chemical residue analysis will provide another angle with which to approach these topics and valuable information.

CHAPTER THREE
METHODOLOGY

"Organic residue analyses performed with the appropriate degree of rigour are essentially forensic analyses" (Evershed 2008a:912). The premise in both disciplines is to work backwards to understand the original scenario (or pottery contents in our case) from the degraded and altered remains that survive. This chapter, with its introduction to lipids, review of methodological literature, and outline of collection, extraction, and interpretation techniques employed in this study, illuminates the ways in which Evershed's observation was accurate.

When archaeologists use the term 'residue analysis', they can be referring to a number of methodologies whose purpose is to separate and identify specific compounds. Residue analysis can refer to starch grain, carbohydrate, protein, lipid, alkaloid, DNA, or other biomolecular analyses (Heron and Evershed 1993). *Theory and Practice of Archaeological Residue Analysis* (Barnard and Eerkens 2007) provides an excellent cross-section of case studies in these various areas. According to Barnard and Eerkens (2007:5), studies employing organic residue analysis have risen from less than 2% of all articles in *Journal of Archaeological Science* and *Archaeometry* in the 1980s to 10% by 2004. Each type of residue is analyzed with specific processes and with varying success rates. This study focuses specifically on analyzing lipids, the hydrophobic molecules that are ubiquitous in organic substances. Lipids are generally less prone to degradation than carbohydrates, proteins, or DNA, a quality that has contributed to lipids becoming the most prominent type of organic residue analysis (Eglinton and Logan 1991:316;

Evershed 1993; Heron and Evershed 1993). Lipid analysis is often referred to generically with the terms ‘chemical residue analysis’ or ‘organic residue analysis’, terms which I also will use interchangeably henceforth.

The prevalence and success of lipid analysis can be attributed to the chemical and physical properties that enhance the preservation of these compounds. This class of molecules includes fats, oils, waxes, steroids, terpenoids, alkanes, etc., whose structures, although all have a carbon backbone with hydrogen atoms attached, vary considerably in structure between linear, branched, and cyclical, and in degree of saturation (Barnard, Dooley, and Faull 2007:42; Eglinton and Logan 1991:318; Evershed 1993:75). They are remarkably stable molecules, since their hydrophobic nature makes them resistant to hydrolysis and subsequent leaching by ground water, and to thermal degradation. Most are fully soluble in organic solvents and are therefore easily extracted from inorganic matrices such as pottery (Brown and Brown 2011:54; Eglinton and Logan 1991:318; Oudemans and Boon 2007:97).

Lipids can preserve in the archaeological record in a variety of ways, but the focus here will be how they preserve in relation to pottery: as vessel fills, visible residues, or absorbed residues (Evershed 2008a; Heron and Evershed 1993). Vessel fills are the actual undisturbed contents of pots, which are very rare. One such example is a lidded tin canister from a Roman temple that preserved its original contents: a face cream made up of animal fat, glucose, and cassiterite that was once heated (Evershed et al. 2004; Evershed 2008a). Visible residues, aptly named, are tangible surface residues left clinging to the interior or exterior of the pot. On the exterior, residues are most often soot resulting from being placed in an open fire (Evershed 2008a; Heron and Evershed

1993:250). However, residues can also form on the exterior from interior contents that have seeped through during cooking and been carbonized by the heat (Evershed 2008a:904). Visible residues are more prone to contamination since they are exposed, but they may be somewhat more protected from microbial decay by "encapsulation and grafting of the compounds inside the macromolecular structure of the char" (Oudemans and Boon 1991:224). Interior visible residues can be caused from scorching a meal during a cooking episode, waterproofing a vessel for storage or transport of goods, or other industrial uses (Beck et al. 1989; Evershed 2008a; Mills and White 1989). The heating itself can alter the compounds (Oudemans and Boon 1991:225). Absorbed residues, far more common archaeologically, refer to organics that have been incorporated into the pottery matrix as a result of the use of a pot (Evershed 2008a; Heron and Evershed 1993). They can only be analyzed by chemical extraction from the pottery matrix. Absorbed residues typically represent the cooking or processing of foodstuffs, but can also survive from any non-culinary use that aids in transferring lipids into the vessel walls (Evershed 2008a; Heron and Evershed 1993:251). Evershed estimates ">80% of domestic cooking pottery assemblages worldwide" contain absorbed residues (Evershed 2008a:904). The pottery matrix works as a protective barrier to preserve the organic molecules from decay and microbial activity. Microbial access is thought to be limited, because of the small pore size of the pottery (Evershed 1993:77; Pollard et al. 2007:148). As Eglinton and Logan (1991:320-321) explain, "Any physical barrier reduces the availability of electron acceptors and nutrients required for microbial decomposition." However, the pottery matrix itself can cause structural changes to occur when organic compounds interact with minerals in the clay, especially in the presence of heat. For

example, experimentation has shown that long chain ketones with 31, 33, and 35 carbons are formed from heating fatty acids, specifically $C_{16:0}$ and $C_{18:0}$, and triacylglycerols above 300° C in clay containers (Evershed et al. 1995; Raven et al. 1997).

The organic compounds that survive in archaeological residues are usually altered forms of the original compounds (Evershed 1993, 2008a; Evershed et al. 1992; Heron and Evershed 1993; Mills and White 1994; Morgan et al. 1973; Thornton et al. 1970). Identification is frequently based not on the parent or original compound itself, but on its breakdown products (Brown and Brown 2011; Evershed 1993; Mills and White 1994). An understanding of "molecular taphonomy", as Eglinton and Logan (1991:320) refer to it, is a must when interpreting residue results. Initial degradation seems to be rapid, after which the molecules remain relatively stable when compared to other biomolecules, but are still not immune to further degradation (Brown and Brown 2011). In Dudd, Regert, and Evershed's (1998) experiment, there was a >95% reduction of TAGs from milk fat after just 25 days and a >84% reduction of TAGs from olive oil after 20 days. Reber and Evershed (2004) experimentally showed that maize lipids rapidly degraded to the point at which they could not be securely identified to this foodstuff after six months of burial and a mere three months for vessels that were used for only one maize cooking episode. Long term degradation continues and different compounds decay at different rates, which can lead to recovery bias. Some general trends of preservation are noted below. Saturated fatty acids tend to preserve better than unsaturated fatty acids (Evershed et al. 1992; Heron and Evershed 1993; Mills and White 1994; Rottländer and Schlichtherle 1983). Within unsaturated fatty acids, the higher number of double bonds or higher degree of unsaturation tends to decrease its stability; they are more likely to be affected by

polymerization, oxidation, and microbes (Evershed 1993:85; Evershed et al. 1992; Mills and White 1994:34; Rottländer and Schlichtherle 1983). Fatty acids with one double bond preserve better than polyunsaturated acids, those with two or more double bonds (Heron and Evershed 1993; Rottländer and Schlichtherle 1983). Therefore, the characteristic polyunsaturated fatty acids from marine resources and some plants have an overall lower preservation potential (CoBabe and Pratt 1995; Guitart et al. 1999). Long chain acyl compounds tend to preserve relatively well because of their lack of functional groups and usually saturated forms (Evershed 1993:85). Even though sterols and hydrocarbons are generally more impervious to decay than fatty acids, they too can be oxidized (Evershed 1993:80).

A major issue within residue analysis that must be taken into account is contamination. From the moment the pot is discarded from use, it is subject to contamination. Initially, it was argued that the organics found in residue results could be inherent in clay, questioning the validity of absorbed residues (Heron and Evershed 1993:256). Although organics are present in clay or can be intentionally added as temper, they are mostly burned off with the high heat used to fire the pot (Heron and Evershed 1993; Johnson et al. 1988; Reber et al. 2018). Post depositional contamination may originate from the burial environment itself (Heron et al. 1991). When the quantity of lipids is very low in the sherd to begin with, the relationship with the soil is unclear (Heron and Evershed 1993:255; Heron et al. 1991:655-656). Ideally, soil samples are taken with sherds during excavation to test for post-depositional contamination (Heron et al. 1991:657). However, in the event that the material was previously excavated and soil samples were not saved, handles or kiln wasters can be sampled as proxies for the amount

of background soil lipids (Condamin et al. 1976:201; Heron and Evershed 1993:256). Contamination may also be modern, meaning that it occurred post-excavation. This contamination comes from the handling of the pottery in the field, in storage, and in laboratory environments (Evershed 1993; Evershed et al. 1992; Oudemans and Boon 1991). Natural oils from human skin as well as dirty hands that may have recently been touching food, coffee, or tobacco (to name a few often found) can contaminate pottery during handling. The combination of cholesterol and squalene, both found in human skin, is a good indication of modern handler contamination (Evershed 1993:90). Even modern lotions and DEET from bug spray can show up in results (Reber et al. 2015; Reber 2017). Plasticizers are also main contaminants from the modern environment; highly durable plastic molecules can overshadow trace amounts of archaeological compounds (Evershed 1993; Oudemans and Boon 1991). Straight soil-to-laboratory procedures for vessels that will undergo residue analysis can combat modern contamination (Heron and Evershed 1993). Although certainly ideal, this limits residue analysis to only current, ongoing excavations. Given that chemical residue analysis became well established only in the 1990s, there is a backlog of previously excavated material that could undergo residue analysis. Careful laboratory work and analyses can tease out any post-depositional contamination that is present in previously excavated artifacts. Considering how ubiquitous lipids are, every avenue for contamination must be considered (and reconsidered), much to the headache of the researcher, in order to have confidence in the results.

Regardless of these limitations, chemical residue analysis is a useful tool. It can provide direct evidence of vessel function, which is rarely achieved by other

archaeological analyses (Charters et al. 1993, 1995; Heron and Evershed 1993). The use of vessels is usually indirectly inferred based in contextual information. It can shed light on methods of cooking, such as boiling, roasting, etc., or reveal manufacturing or maintenance techniques of pottery, such as waterproofing or repair. It can reveal biomarkers, compounds or suites of compounds that link to a specific genus or species (Evershed 1993, 2008a; Evershed et al. 1990). It can reveal ephemeral plant remains in the archaeological record that would remain undetected, such as leafy greens or spices, or liquid foodstuffs. Insomuch as bones and to a lesser extent, macrobotanicals survive in the Mediterranean, only in exceptional preservation environments would liquid remains preserve. When residue data is integrated with the paleoethnobotanical and zooarchaeological record, a more complete picture of plant and animal exploitation in the past can be created. It can even confirm pigment sources, such as the famed Royal Purple dye of antiquity that is made from mollusks (McGovern and Michel 1984). To appreciate the importance of this tool to archaeology, the mechanics and history of the method will now be discussed.

History of Residue Analysis: Method Development and Applications

The invention of gas chromatography (GC) in the mid-twentieth century within the discipline of analytical chemistry and its subsequent linkage to mass spectrometry (MS) twenty years later provided the foundation for organic residue analysis to emerge (Bartle and Meyers 2002; Evershed 2008a:896). Highly sensitive instrumentation had to be developed to detect the often trace amounts of lipids that are preserved before it could be applied to archaeological samples (Roffett-Salque et al. 2017:627). It is estimated

through experimentation that only about 1% of the original lipid content survives through archaeological time (Evershed 2008b:28). Fortunately, GC/MS is sensitive enough to detect as little as 1pg of lipid (Brown and Brown 2011:62). Chemical residue analysis of lipids has been a tool utilized in archaeology for nearly 50 years. No longer in its infancy, the field has experienced its fair share of growing pains with expectations and limitations and has now matured with more refined methods and clear strategies for interpretation.

It is important to outline the principles behind GC/MS before proceeding to a discussion of method development for archaeology. In short, GC separates complex organic mixtures into their smaller constituent compounds, while MS produces a mass spectral signature of each compound that allows for identification. Upon injection into the GC, the sample is immediately vaporized into a mobile gas phase and then it moves through the stationary phase, which is coated on the interior of the chromatography column. The stationary phase varies from column type with respect to thickness, chemical makeup, and polarity, which effects the resolution of compounds (Kitson et al. 1996). The column is a thin, hollow tube that is coiled inside the oven through which molecules successively move as the oven temperature rises. The rate at which the compound moves through the column is related to its molecular weight, boiling point, and volatility (Brown and Brown 2011:63). Generally, the low molecular weight compounds elute first while the higher molecular weight compounds elute last. Compounds are measured once they leave the column to produce a gas chromatogram, or graph of peaks showing retention times and relative abundances. The length of time the compound is retained in the column is the first clue in determining the identity of a compound. Once the compound leaves the column, it enters an in-line mass spectrometer.

The mass spectrometer blasts compounds with energy, which causes them to lose an electron, become unstable, and fragment into their molecular ions, M+, as well as a characteristic set of smaller ions. Molecules tend to fragment in predictable ways and it is these fragmentation signatures that are used for identification, the specifics and problems of which are described later in this chapter.

Lipids are most often analyzed with the combined technique of high temperature GC/MS now, but this was not how the field started out. Although not exhaustive, the following discussion serves to highlight important studies that shaped the field of organic residue analysis. The two main trajectories in residue studies will be discussed: methodological and experimental studies and biomarker studies.

Methodological and Experimental Studies

Thornton and colleagues' 1970 paper marked the first use of chemical residue analysis in archaeology to identify lipids, where the authors investigated the fatty hoards found in peat bogs in Scotland and Ireland known as 'bog butters' (Thornton et al. 1970). They used thin layer chromatography (TLC) as a physical means to separate compounds of eight bog butter samples alongside modern butter samples and then submitted them to GC alone to determine fatty acid composition. They identified compounds by comparing their retention times to those of fatty acid standards. They identified bog butters as adipocere, fatty materials that have been degraded by microbial action within an anaerobic environment, based on the presence of high levels of $C_{16:0}$ palmitic acid and low levels of unsaturated $C_{18:1}$ oleic acid (Thornton et al. 1970:21). This study was important because it was the first time archaeological lipid samples had been chemically

characterized and shown to preserve as degraded forms. They argued that in order to identify the origin of archeological compounds, one must account for the considerable degradation that has happened and exercise caution when determining the parent compound (Thornton et al. 1970:24).

In 1973, Morgan and colleagues confirmed experimentally Thornton and colleagues' (1970) findings. They conducted an experiment to see if butter left in the waterlogged environment of a peatbog would degrade into compounds resembling adipocere. They tested the experimental samples at intervals over the course of two years with GC. They found that it did degrade into an adipocere-like substance with the decrease in $C_{18:1}$ and the increase in $C_{16:0}$ (Morgan et al. 1973:9). They confirmed that considerable alteration of the compounds had occurred and traditional methods of identification could not be used to identify these altered compounds (Morgan et al. 1973:10).

Research by Condamin and colleagues (1976) tested the hypothesis that a class of Mediterranean amphorae once contained olive oil and that remains could be preserved in the vessel walls. They were the first to suggest that organics could have seeped into the porous pottery matrix and the first to attempt extraction of these residues. They tested various solvents and found that chloroform and methanol provided the most efficient extractions (Condamin et al. 1976:196). They collected 100 g samples from 20 amphorae found in terrestrial and underwater excavations that were thought to have contained olive oil. They halved each sample into two lateral parts to test the interior lipids versus the exterior lipids to see if a concentration gradient could be observed. Identifications were made by comparing only peak retention times on some samples and GC/MS on others

with a packed column. They tested for fatty acid composition only and found that concentrations were eight times the amount internally as externally, which they interpreted as evidence of olive oil seepage into the vessel walls (Condamin et al. 1976:199). They also tested the soil when it was still attached to the amphora wall, as well as rejects from a pottery kiln that would not have contained anything (Condamin et al. 1976:197). They found some similarity between the amphora samples and soil, but the soil lipids were found in considerably lower quantities. Later, it was pointed out that they were not bringing the temperature high enough to capture all of the compounds and furthermore, that their processing protocol likely caused data loss of the intact large molecular weight molecules (Heron et al. 1991:643). Although there were flaws, this was a quite sophisticated study for its early date. The authors compared soil samples to pottery samples, investigated differences between internal and external lipid amounts, used kiln wasters as controls, and tested matrices for absorbed residues, although the latter advancement did not gain traction until at least a decade later.

Over the next decade, several more applications of lipid analysis to archaeological specimens were published, some which were still limited in scope, but all building on the budding successes of the previous decade. Two studies were published in the 1983 *Symposium on Archaeometry Proceedings* that focused on preservation of lipids in pottery. Evans and Hill (1983) tested visible residues from seven sherds from Danish Neolithic sites using successive solvents, TLC, and high-performance liquid chromatography and found linseed oil, pork fat, bird or rabbit, and cooked eggs via protein analysis (Evans and Hill 1983:224-225; 227). Rottländer and Hartke analyzed 10 small serving bowls from the site of Noreia using GC and found that they held degraded

olive oil, poppy oil, pork fat, and fish (Rottländer and Hartke 1983:218). They noted that although decomposition had occurred, relative percentages of fatty acids can be used to identify substances (Rottländer and Hartke 1983:219). Both studies based species-level identifications on fatty acids alone, which would later be questioned and discounted.

Lin and colleagues (1978) applied residue analysis to a different data set--coprolites. They studied six coprolites dating to 50 BC-AD 100 from Lovelock Cave, Nevada. They performed TLC to separate neutral compounds from the bile acids and submitted all resulting bands to gas-liquid chromatography and mass spectrometry (GLC/MS) using a short U-shaped packed column. They were able to quantify lipids, since they added a standard (Lin et al. 1978:217). Cholesterol, campesterol, stigmasterol, and β-sitosterol, as well as their derived stanols and stanones were found in both the coprolites and fresh stool samples, while the bile acids were found in lower quantities in the coprolites. Surprisingly, nearly a quarter of the sterols in the coprolites was comprised of unmodified cholesterol, whose preservation was attributed to the desiccated environment. They compared the sterol content to the stools of eight living Tarahumara Native Americans with high and low cholesterol diets and found that the steroid content was comparatively lower overall in the coprolites, confirming some degradative loss. The authors cautioned against using absolute quantities in lipid comparisons, because the rate of degradation cannot be measured. The ratios of bile acid to cholesterol and plant sterol to cholesterol showed considerable variation from one coprolite to another; they argued that the food supply was uncertain during the occupation of the cave (Lin et al. 1978:220). This analysis, although somewhat clunky in its methodology, was headed in

the direction towards asking more substantive questions of the data about diet and resource availability.

Morgan and colleagues (1984) collected four samples from a Thule frozen midden at the Washout Site in the Western Arctic and submitted them to TLC and GC with a packed glass column. Various seal species were identified in the faunal remains, but the authors suspected that whales were also consumed by the Thule. A large percentage of gadoleic acid ($C_{20:1}$), indicative of a marine source, was identified in all samples. Fish was ruled out, because of the lack of polyunsaturated fatty acids, which they argued should have survived with the level of preservation at the site. The authors compared the fatty acid composition of the samples to that of modern whale, seal, and walrus species and concluded that the midden fats were a mixture of seal and whale (Morgan et al. 1984: 46). They argued that lipid analysis can contribute to our understanding of subsistence patterns and reveal the utilization of species that were not represented in the zooarchaeological record. Again, these conditions were targeted and believed to have only been successful because of the frozen, anaerobic environment.

Tarry substances from shipwrecks were investigated in a later study and the first unambiguous biomolecular identification of pitch and tar was made via chemical residue analysis (Evershed, Jerman, and Eglinton 1985; Robinson et al. 1987). Samples from the ship's structure, barrels, tarred rope, and a tar solid were taken from the sixteenth century *Mary Rose* and a black substance thought to have leaked from an amphora from a seventh century BC Etruscan shipwreck. The archaeological samples, modern pitch, and modern tar samples were subjected to a litany of analyses: elemental composition, infrared spectroscopy, nuclear magnetic resonance spectroscopy, GC, and GC/MS (Robinson et

al. 1987:638). The *Mary Rose* and the Etruscan samples were very similar to modern pine manufactured Stockholm tar, based on the presence of diterpenoids, namely retene, dehydroabietic acid, and methyl dehydroabietate. The authors briefly discussed steps taken to minimize contamination, such as doubly distilling both the water and chemicals used in processing. This comparison of methods showed how GC/MS was most informative in revealing the chemical signature of samples, but authors pointed out the difficulty in interpretation and the labor-intensive chemical processing. This conclusion was independently confirmed a few years later by Beck and coauthors in their comparative study of transport amphora residues from ancient Carthage (Beck et al. 1989). GC alone could not provide enough information to characterize the samples fully. Anaerobic conditions of shipwrecks provided the means to preserve these samples, although Mills and White (1989) had pointed out that lipids would likely survive in terrestrial sites as well. Beck and colleagues' study demonstrated the first biomolecular characterization of non-food residues in pottery.

Evershed and Connolly (1988) successfully recovered lipids from the 2,000-year-old bog body, so-called "Lindow Man", expanding the limits of the type of materials that can preserve lipids. They analyzed a psoas muscle sample and found highly degraded triacylglycerides, phospholipids, and cholesterol. They found little correlation between the surrounding peat and the muscle tissue, suggesting that very little, if any, migration of environmental lipids had occurred.

The 1990s witnessed a burgeoning of organic residue analyses in archaeology and a much-needed emphasis on scientific rigor and experimental testing of contamination, degradative pathways, and interpretations. At the beginning of the decade, a volume

based on a symposium in 1989 entitled *Organic Contents of Ancient Vessels* was meant to provide a state of the research (Biers and McGovern 1990). Organic pottery analysis was the topic of Beck and Borromeo's (1990) article on pitch manufacturing identification from shipwrecks, Rottländer's (1990) overview on lipid analysis, and Gerhardt and colleagues' (1990) non-destructive extractions of perfume vessels.

One of the many important studies from this decade was Evershed and colleagues' 1990 study detailing a new methodology for residue extraction. Members of this research group would go on to dominate the field even today. They sampled newly excavated pottery sherds from the Raunds Archaeological Project in the UK. Their study used high temperature GC/MS with a capillary column and for the first time extracted a total lipid extract without pre-fractionation by TLC. This allowed the high molecular weight lipids to be recovered—triacylglycerides, diacylglycerides, etc.—because the method did not require saponification first. The type of column and high temperature increased resolution, separation of compounds in a single run, and decreased processing time (Evershed et al. 1990:1340). They suggested using this as a pre-screening tool to see if fatty acid and neutral component analysis is warranted. The capillary column had been first used by Beck and colleagues (Beck et al. 1989). However, it was the combination of the column, the high temperature, and the lack of saponification before submitting samples to GC/MS that was innovative. This refinement of method marked an expansion of the explanatory potential of residue analysis.

Heron and colleagues (1991) conducted experiments to study the migration of soil lipids into sherds. They used 10 newly excavated Late Saxon/Medieval sherds from the West Cotton Site, UK, with soil still attached. They analyzed the soil adhering to the

sherd, a sample of the sherd itself, and three soil samples from the site. The amount of lipid in the soil was found to be far less than in the sherds. All three soil samples were shown to be chemically similar, with the most abundant compound being a long chain fatty acid, pentacosanoic acid ($C_{25:0}$), which is rare in most foods (Heron et al. 1991:646, 655). The soil scraped from the sherds also matched the characteristic "fingerprint" of the three soil samples (Heron et al. 1991:649). Very low abundance of pentacosanoic acid and high abundance of significantly different lipid profiles in the sherds supported their hypothesis that the movement and absorption of soil lipids into buried pottery sherds is minor (Heron et al. 1991:655-656). Only one sherd/soil pair matched suggesting interaction, but this sherd's lipids were in very low concentrations when compared to the other sherds. The authors suggested that there is a background level of sherd lipids that are present in the adhering soil, but when lipid levels are high, they swamp out any background comparisons (Heron et al. 1991:656). Migration of soils into the ceramic matrix and microbial activity were found to be negligible for the first time experimentally.

A year later, Evershed and colleagues (1992) brought up an issue to which surprisingly little attention had been paid—the presence of modern contamination in ancient residue results. They discussed measures that must be taken to prevent or decrease contamination, setting the standard for what is considered best practice now. Up to this point, contamination was rarely discussed in studies and likely not considered. The previous studies on the *Mary Rose* pitch/tar samples had only briefly mentioned contamination (Evershed et al. 1985; Robinson et al. 1987). To minimize contamination, they recommended wearing gloves when handling pots/sherds to reduce the transfer of

lipids from human hands, cleaning glassware beyond standard cleaning to prevent cross-contamination, using disposable glassware, avoiding plastic laboratory supplies, and using high grade solvents or distilling them (Evershed et al. 1992:191). The realization that contamination can enter samples from so many avenues questioned the validity of the earlier studies and the extent to which lipids reported were authentically archaeologically-derived. They noted also that GC columns coated with polysiloxane provide better resolution. They called for large numbers of pots to be sampled, so as not to just be doing residue analysis for residue analysis' sake. This article was the first to point out how contamination must be a methodological consideration and steps must be taken to reduce the risk.

In another landmark paper, Heron and Evershed (1993) published a critical assessment of residue interpretations. Contrary to the promise of residue analysis, they pointed out the considerable challenge it is to differentiate a mixture of substances used simultaneously in a vessel from separate, independent uses of different substances though time in a vessel (Heron and Evershed 1993:258). They suggested that other lines of evidence such as experimental, ethnoarchaeological data, or comparison of visible residues against absorbed residues of the same sherd could resolve this; residue analysis alone cannot recreate ancient recipes (Heron and Evershed 1993:258-259). They criticized the fact that very little residue research had been conducted to determine function of a vessel, the area where the methodology has the greatest potential to contribute. They argued that using only fatty acids to interpret residues can lead to incorrect and unsubstantiated origin interpretations and called into question the validity of previous studies, because of rates of decay, diagenetic alterations, and the lack of

highly diagnostic fatty acids (Heron and Evershed 1993:269). Many of the previous studies did not detail how their interpretations were obtained, which makes it impossible to assess their validity (Heron and Evershed 1993:268). Heron and Evershed (1993:268) called for them to be rejected.

More experimentation to test archaeological lipid interpretations was undertaken by Charters and colleagues (1993). They tested the hypothesis that analyzing absorption patterns of lipids across pottery vessels can help determine vessel use. They sampled from the base, body, and rim of 62 reconstructed vessels. They quantified residues in each area by comparing the peak areas to the known area of the internal standard. Differential preservation rates were indeed observed from the vessel part, which they argued were the result of unique vessel use among types. They suggested that the accumulation pattern on the jars, with the highest lipid concentration at the rim and body, was the result of boiling foodstuffs, whereby lipids were freed to rise and absorb at the internal water level on the vessel (Charters et al. 1993:218). Another vessel type had the highest lipid concentration on the base, which could point to roasting as the cooking mechanism (Charters et al. 1993:218). They advised that researchers have a good understanding of sherd position prior to residue sampling if the goal is to determine vessel function (Charters et al. 1993:212).

Charters and colleagues (1995) applied the method from their 1993 study to determine the function of two vessels with unusually high lipid concentrations from the West Cotton site in the UK. They sampled from the base, body, and rim of a jar and the base and rim of a bowl (Charters et al. 1995:114). The bases of two vessels were similar to each other with waxy compounds indicating beeswax, and the rims of two vessels were

similar to each other with fatty compounds and cholesterol identified as meat (Charters et al. 1995:119-121). The body of the jar was a mixture of the two (Charters et al. 1995:119). Lipid concentrations, however, were different. They decreased from the rim to the base in the jar, while they were more evenly distributed in the bowl. It is unlikely beeswax was being as a sealant, since beeswax was not found in any other vessels of the hundreds tested. They instead suggested that the substances were added in stages: beeswax was heated, saturating the base and body first, and then meat was added and heated. Meat lipids could only enter the pottery pores where the beeswax had not yet absorbed. The authors then disproved experimentally that a mixture of beeswax and fatty acids would separate into the archaeologically observed pattern upon cooling, thus lending credence to their additive hypothesis (Charters et al. 1995:124). They argued that the sequence of substances added and heated affects their distribution and concentration across the vessel. Scorching patterns consistent with direct heat were observed on the jar and not the bowl, which would account for the low quantity of lipids in the jar and point to different uses (Charters et al. 1995:124-125). Surprisingly, it would not be until the next century that cross vessel sampling would be applied again to determine vessel function (Cramp 2008; Cramp et al. 2012; Reber et al. 2015).

Charters et al. (1997) later conducted experiments on boiling leafy vegetables to test the dispersion pattern that they had proposed in their initial article four years prior. They conducted 10 successive boiling episodes of cabbage leaves in replica pots, sampling the base, body, and rim after each boiling and submitting the samples to GC/MS (Charters et al. 1997:4). After one boiling of cabbage leaves, it took considerably less time to reach boiling temperature by an 80 minute reduction, which suggests that

lipids effectively impregnated the vessel after one use. Higher concentrations of lipids did appear in upper parts of vessel as they had found archaeologically. After hundreds of uses, this pattern would be even more apparent. The boiling water level near the top helps the absorption of epicuticular waxes that have been released from the leaves (Charters et al. 1997:6). Heat from the 800° C fire caused lipids at the base to likely degrade, which is seen in the very low lipid rates (Charters et al. 1997:6).

In Boëda and authors' (1996) study, one of the oldest visible residues ever detected via GC/MS was identified from a Levallois flake and a scraper. Found at Middle Paleolithic site of Umm el Tlele in Syria, the flake and scraper both had a black substance that appeared to be the outline of handle attachments. The authors isolated alkanes and aromatics from C_{15} up and found a molecular profile similar to once heated bitumen, although one that was chemically dissimilar to well-known sources in the region (Boëda et al. 1997:337-338). This study extended the preservation of archaeological, non-fossilized lipids back to 40,000 years ago.

Stern and colleagues (2000) investigated concentration gradients, as a means to assess depositional and modern contamination. They conducted a study of eight Late Bronze Age Canaanite amphorae found in Amarna, Egypt, believed to have transported vegetable oils. They tested various methods of residue extraction and found that standard solvent extraction with saponification afterwards yielded the highest overall lipid content (Stern et al. 2000:412). They sampled in 2 mm cross-sections throughout the width of the sherd (Stern et al. 2000:402). They confirmed concentration gradients in six of the eight of samples. The largest concentration of lipids was found in the first 2 mm of the inner surface, the least in the core of the sherd, and a larger amount in the outer side, but in a

lesser quantity than the interior (Stern et al. 2000:412). They used these findings to argue against too aggressively cleaning the sherd prior to sampling as it would decrease lipid recovery. In the event that samples were handled post-excavation and no soil samples were saved, sampling throughout the sample wall could assess the amount of contamination. This would seem to suggest that the external lipids could be subtracted from the internal lipids to reach the amount of archeological lipid, although the authors did not explicitly state this. They detected no contaminants in the amphorae. Their cross-sections were 0.1 g in weight, the smallest amount of pottery from which lipids had been recovered (Stern et al. 2000:412).

Evershed and coauthors (2002) argued that determining the origin of animal fats can be problematic, even though identifying the fatty acids and sterols comprising them is relatively routine. This was demonstrated with the organic residue analysis of so-called 'dripping dishes' and lamps. The ratios of $C_{16:0}$ to $C_{18:0}$ were different between the two vessels; the lamps also had more odd carbon chain length unsaturated fatty acids and more branched fatty acids (Evershed et al. 2002:664). However, these alone cannot identify different origins of the fats, especially since some of the smaller chain fatty acids are easily degraded and are rarely recovered archaeologically (Evershed et al. 2002: 665). However, incorporating stable isotope analysis showed that the fats in the dripping dishes were from monogastric animals, while the lamps were from ruminant animals (Evershed et al. 2002:664). This article showed the limitations of using fatty acid ratios and the benefits of incorporating stable isotope to identify animal fat origins.

Reber and Evershed (2004) conducted experiments to determine the effect of burial environments and cooking episodes on lipid preservation, focusing on starchy

foods. Starchy foods tend to be underestimated in lipid studies, because when cooked with other high lipid foods, they are easily overshadowed and no biomarkers are known (Reber and Evershed 2004: 400). A starch with a higher lipid content than most, maize, was used in this study (Reber and Evershed 2004:401). Whole kernels, coarse cornmeal, and fine cornmeal were cooked in separate pots for four hours and then sherds from these pots were left in different environments: indoors, on the ground surface, buried in loamy clay, and buried in clay loam (Reber and Evershed 2004:401-402). Maize kernels and coarse cornmeal were cooked in another pot in repeated episodes over several months and then placed in the same burial contexts (Reber and Evershed 2004:402). All pots were left in their context for six months and then subjected to GC-MS analysis. The only pot that preserved any residues after six months was the pot used to cook multiple maize meals (Reber and Evershed 2004:403). Reber and Evershed argued that the pots used only in one cooking episode yielded no lipids, because of the low relative lipid content in maize, which rapidly decomposed or leached out in pottery, likely after only three months (Reber and Evershed 2004:407). No biomarker for maize could be established. They argued that the observed underestimation of lipids from starchy foods should be accounted for in residue interpretations. The environment in which pottery is buried has an effect on the abundance of fatty acids, both unsaturated and saturated (Reber and Evershed 2004:404). Lineoleic acid ($C_{18:2}$) preserved only in the room temperature sample; oleic acid ($C_{18:1}$) did not preserve well indoors, but did preserve well in clay loam (Reber and Evershed 2004:404). Longer-chain saturated fatty acids were not altered as much by burial environment. They experimentally confirmed that depositional environment affects fatty acids differently.

An eye-opening blind round-robin experiment demonstrated how identifications and interpretations are not as straight forward as they may seem (Barnard, Ambrose, et al. 2007). An experiment was conducted to assess methods for correctly identifying organic residues from ceramics and gauge variation between labs (Barnard, Ambrose, et al. 2007). Camel milk and mineral water were steeped in an unglazed earthenware bowl from Egypt for 24 hours, cooked and cooled twice, and then left to air dry for ten days. Lengthwise strips of the pot were doled out to several labs without any context. The main compounds expected by residue analysis were stearic ($C_{18:0}$) and myristic acids ($C_{14:0}$) with minor amounts of their mono-unsaturated forms. Casein should be found if protein analysis was performed, while stable isotope analysis should have revealed a mixture of C4 and C3 plants. Two labs found no residues. The other labs found evidence for veal, egg, or goat milk (lab B); decomposed roots, tubers, or berries (lab C); veal or egg (lab G); milk of an herbivore (lab J); and an animal product (lab K) (Barnard, Ambrose, et al. 2007:33-34). Only a few labs conducted protein analysis and stable isotope analysis. Surprisingly, the closest interpretation to the actual contents was from stable isotope analysis from one of the labs (Barnard, Ambrose, et al. 2007:31). This work highlights the difficulties in pinpointing the origin of residues down to a specific animal or plant, especially when there is no context. The authors discussed how residue analysts must combine residue data with other lines of evidence for a full interpretation. The region and animal/plant distribution needs to be taken into consideration, so that the analytical tools can be tailored.

In 2008, Evershed authored two important articles: one where he discussed some of the short-comings of the field, and a second where he discussed experimental

approaches to organic residue analysis (Evershed 2008a; 2008b). In the first, he argued that paleoenvironmental information should be incorporated into organic residue analyses (Evershed 2008a). Only domesticated plants and animals should be considered, since they have the highest likelihood of having been used often enough to have left residues (Evershed 2008a:899). Since most fatty acid profiles contain only undiagnostic compounds, stable isotopic ratios should be employed as a complementary analysis. He argued that recognizing altered forms of original biomarkers are crucial, since the original degrades over time (Evershed 2008a:900). He critiqued that a large amount of non-peer reviewed studies appeared early on, up to 1990s, that "misled the archaeological community as to the analytical chemical rigour required for meaningful results" (Evershed 2008a:896). In the second article, Evershed (2008b) gave an overview of the various experiments that had been performed to better understand lipid behavior. He remarked that experiments should be driven by archaeological observations of lipid behavior to test interpretations, not the reverse.

On a side note, all the techniques discussed so far have been destructive techniques, in which part of the pottery is irreversibly consumed in the process. There have been a few non-destructive techniques that have claimed success: Gerhardt and colleagues (1990), Koh and Betancourt (2010), and Vanderveen (2011). However, as Heron and Evershed pointed out over twenty years ago and remains true at present, none of these methods have been experimentally confirmed or rigorously tested as to their validity (Heron and Evershed 1993:264).

Biomarker Studies

Most of the above studies have been methodological in nature, but biomarker studies are another avenue of research. Since the first study using the biomarker approach was published, many other studies have focused on identifying biomarkers or have stumbled upon them in research. In 1983, Knights and coauthors investigated Roman military diet through chemical analyses of a defensive ditch fill in Scotland (Knights et al. 1983). It was argued, based on pollen, macrobotanicals, and other lines of evidence that the ditch was filled by sewage from the nearby latrines; however, no human coprolites were found (Knights et al. 1983:140-143). Lipid analysis was conducted on soil samples by chloroform/methanol extraction. Samples were saponified, fractioned into non-saponifiable and acid fractions, and then fractioned further with TLC; they were finally submitted to GLC and then GC/MS in packed columns (Knights et al. 1983:145). Coprosterol, formed from bacterial action on cholesterol in human intestines, Δ_5 sterols, 5α-stanols, and deoxycholic acid provided strong evidence of fecal presence in the bottom layer of the fill. This suggested to the authors that the ditch was being filled by drainage from the latrines and that the Roman soldiers ate primarily a plant-based diet (Knights et al. 1983:149). This was the first study to use the biomarker approach of lipids, here using sterols and bile acids to identify human fecal inputs. It would not be until 1993 that this approach would be formalized.

In the late 1980s, two studies focusing on different materials were published. Rullkötter and Nissenbaum (1988) analyzed black coatings from Egyptian mummies and coffins to search for the presence of Dead Sea asphalt in mummification. They found that three were identical to the Dead Sea asphalt with a sequence of long, predominantly odd

chain n-alkanes, sterane, and triterpanes, while the oldest sample had no n-alkanes and abundant diasteranes, likely from a different crude oil source (Rullkötter and Nissenbaum 1988:619). Crude oils and asphalts have very stable molecules with highly distinctive biomarkers that are easily detected with only trace amounts. Mills and White (1989) tested amphorae resins from the Ulu Burun shipwreck and used the biomarker approach to determine that the resins were not produced from pine as previously found at other sites. The Ulu Burun samples were identified as Chios turpentine, *P. atlantica*, because of triterpenoids and the lack of diterpenoids characteristic of conifer resins (Mills and White 1989: 40).

As part of a large-scale residue analysis research program of the Raunds Area Project, Evershed, Heron, and Goad analyzed five sherds and found distinct lipid profiles composed of nonacosane, nonacosan-15-one, and nonacosan-15-ol thought to belong to epicuticular waxes of higher plant leaves (Evershed et al. 1991:541; Kolattukudy 1976). The authors tested modern cabbage leaves known to have these compounds, and confirmed that they were present in the same abundance as in the five sherds (Evershed et al. 1991:541). This suggested that the pots were used to cook cabbage, which was the first time leafy greens had been confirmed by any analytical means in archaeological samples. They proposed this suite of compounds as biomarkers for cabbage (Evershed et al. 1991:543)

Evershed (1993) authored a thorough overview of lipid analysis in archaeology on the heels of the popularization of ancient DNA studies. He coined the term biomarker, borrowing from paleontological studies, and introduced the concept as a way to link compounds or suites of compounds to contemporary plants and animals that may have

been used in antiquity; he discussed biomarkers that are possible within various lipid classes (Evershed 1993:78-79). He advocated more experimentation to better understand decay and to identify important biomarkers, in addition to an expansion of reference databases to better align with the plants and animals that may have been employed in the past (Evershed 1993:90).

Many more biomarker studies began to appear, but for brevity's sake only a few more of these studies will be highlighted. Heron et al. (1994) characterized a black residue from a Neolithic potsherd found at Ergolding Fischergasse in Germany with high temperature GC and GC/MS. They found that the sherd contained components found in beeswax, mostly even number wax esters from C_{40} to C_{50}, but did not find the other characteristic compounds that were characteristic of beeswax, such as odd numbered n-alkanes (Heron et al. 1994:267). They confirmed experimentally that the complete loss of alkanes could have resulted from the heating of beeswax. This is marked as one of the earliest confirmations of beeswax and suggested hot wax was added for waterproofing or being held for other uses (Heron et al. 1994:268). Although not the first time that beeswax was identified, the importance of this study is in its experimentation to determine why expected biomarker compounds were missing.

Hansel et al. (2004) proposed additional biomarkers for the processing of marine commodities by testing 31 sherds from a coastal site in Brazil with abundant marine remains. They found a series of ω-(o-alkylphenyl)alkanoic acids 16, 18, and 20 carbons in length that eluted after $C_{18:0}$. They argued that these isomers were thermally created from polyunsaturated fatty acids $C_{16:3}$, $C_{18:3}$, $C_{20:3,}$ which are common in the lipid profile of fish (Hansel et al. 2004:3000). The combination of these isomers with two isoprenoid

fatty acids, 4,8,12-trimethyltridecanoic acid and 3,7,11,15-tetramethylhexadecanoic acids, which were already known, provides even stronger evidence for marine resources (Hansel et al. 2004:3000). These could be instrumental in the identification of rarely recovered marine resources.

Reber and Kerr (2012) experimentally tested black drink, a beverage drank by Southeastern Native Americans made from yaupon holly leaves, to determine if biomarkers exist and preserve in burial contexts. The suspected biomarker was caffeine, but the authors hypothesized that it would not preserve in buried sherds because of its high water solubility (Reber and Kerr 2012:2313). They produced five pots from local clay and brewed 20 batches of yaupon holly tea in each, after which they buried potsherds in different soils for two months. Caffeine preserved well in all contexts, while other compounds found in yaupon holly tea did not (Reber and Kerr 2012:2316). They compared the sherd residues to soil samples and no compounds were found to have washed out into the soil (Reber and Kerr 2012:2316).

Summary

Chemical residue analysis has certainly continued to be employed with studies in both of the subareas emphasized here. It has splintered further into whole bodies of literature focusing on regions, time periods, or into the search for specific substances, such as alcoholic beverages. Over the course of 40+ years of lipid analysis in archaeology, the field has grappled with methodological and theoretical challenges. There have been a few key trends that are worth noting. Initially, there was a focus on searching for lipids only in rare preservation environments, such as those with anaerobic

conditions—waterlogged or frozen sites, peat bogs, or underwater—or desiccated environments where the preservation of all artifacts or ecofacts is exceptional. This shifted almost completely the other direction with a realization that residues can and do survive in ordinary environments and are more common than previously thought. Simultaneous with this expansion of environmental loci has also been an expansion of our understanding of the types of media on which lipids can survive: from the early bog or ditch fills to coprolites to bog bodies to pottery to lithics. There has been a dramatic reduction in sample sizes needed for GC/MS, which can surely be attributed to the increased resolution of the instrumentation. The 100 g once used is unheard of these days with the successful lipid extraction from as small as 20 mg (Condamin et al. 1976; Oras et al. 2017). This makes it easier to get permission to sample pottery from often very strict foreign governments. The field has also witnessed an expected refinement and streamlining of methodologies toward dropping off extemporaneous techniques, such as TLC before GC/MS, using capillary columns with higher resolutions and higher temperature limits, and eliminating needless chemical processing before running the initial sample. The latter two advances have significantly expanded the range of lipids that can be recovered. Even more recently, Correa-Ascencio and Evershed (2014) developed a new method that merges extraction and methylation of FA into the same step and increases lipid yield. It is shaping up to be a major advancement in the field. Lastly, there has been an overall trend towards sampling larger numbers of pots in studies and increased scientific rigor, so that we may go beyond mere reporting of organics found and ask more substantial questions of human behavior with the data as well as have more confidence in the results of the analysis.

This discussion has attempted to show how chemical residue analysis developed into a viable tool for archeologists and to provide a framework within which this study can be positioned. I now discuss the methods I employed to study the Ayia Triada pottery.

Methods Employed in this Study

Pottery Selection

The Ayia Triada pottery collection is housed at the Ephorate of Palaeoanthropology and Speleology in Athens, Greece. Some of the pottery is currently stored inside the building, while most is stored outside year-round on a covered, but open rooftop terrace. I noted the location where the vessels and sherds in this study were stored, to determine if modern storage conditions had any effect on the quantity or quality of residue preserved. All of the pottery I sampled was found in Trenches 4, 8, 9, and 11 from Levels 4, 5a, and 5b, the trenches and levels associated with the human burials, grave goods, and the burnt organic layers. These stratigraphic levels were dated to the Early Bronze Age II (EBA II) period by radiocarbon dates and diagnostic pottery (Mavridis and Tankosić 2016a). The classes of pottery found within these levels can be characterized broadly into serving and storage vessels. To date, only a small portion of the more diagnostic and complete vessels have been studied. The rest of pottery from Ayia Triada has not been formally analyzed or published yet.

I sampled from both serving and storage vessels when there was enough of the vessel intact to determine vessel type. I collected pottery samples from sauceboats and bowls of various sizes with everted rims within the serving class and large storage jars

and one lidded container within the storage class. Bowls with everted rims and large storage jars are the two most frequently found types of pottery at Ayia Triada (Mavridis and Tankosić 2016a) and are subsequently the largest proportion of known vessels within my sample set. All pottery samples were taken from the interior of the vessel. Base, body, and rim from the same vessel were sampled when the areas were intact to sample. I also collected a number of samples from base, rim, and body sherds whose vessel type was unknown to me. Although not ideal, it was unavoidable in this study since most of the pottery is still awaiting formal analysis by pottery specialists and is too fragmentary for me to determine their type. It was important to expand the study to include these, however, in order to get a large enough sample size. Handle samples were collected as well, since they can possibly serve as a control to measure the level of background lipid, if any, that existed in the burial environment (Heron and Evershed 1993:256).

Collecting Samples

I completed preparatory work at the Malcolm H. Wiener Laboratory of the American School of Classical Studies at Athens. Due to antiquities regulations, the pottery could not be moved to the Wiener Laboratory for sampling. Samples had to instead be collected at the Ephorate of Paleoanthropology and Speleology, a facility without fume hood or wet lab facilities. High-purity chemicals were obtained in Greece based on local availability. Acetone (ACS Reag. Ph Eur; Merck Chemicals), methanol (Analytical ACS Reag. Ph Eur; Scharlau) and dichloromethane (stabilized with amylene; PA, ACS Grade; Panreac) were used. New disposable glass pipettes and glass scintillation vials were obtained in Greece. I prepped the scintillation vials to receive

samples with a quadruple solvent rinse (dichloromethane/methanol 2:1; v:v). I baked any aluminum foil that would come in contact with the pottery for four hours in a furnace at 400°C, in order to burn off any contaminants that could be present. The baked aluminum foil sheets were sealed in a baked aluminum foil packet and placed in a paper bag for transport to the Ephorate.

I employed a number of other controls during sample collection to minimize contamination. I handled all vessels, sherds, and sampling glassware with disposable nitrile gloves at all times, which I changed between samples to prevent cross-contamination. I regularly created laboratory blank vials into which no ground pottery was added. Blanks were processed exactly the same way from start to finish as the genuine samples and were used to check for modern lab contamination. Only glass lab supplies were used to process the samples to reduce plastic contamination. Prior to taking the sample, I removed a thin layer of the interior of the sherd and discarded it. It is presumably contaminated from post-excavation handling and should not be included in the sample. According to Stern and colleagues (2000), most of the absorbed lipids reside in the innermost 2 mm, so I was careful not to go too deep. All reusable glassware was washed with Alconox and distilled water, dried, and baked for four hours in a furnace at 400°C. Anything that touched the sherd, sample, or solvents was washed and baked, or purchased new, from the aluminum foil to the beakers.

I used an electric Dremel tool with a removable bit to grind off and collect a portion of the sherd. I cleaned the bit before and after each sample to prevent cross-contamination using the following multi-step process. I scrubbed the bit with a disposable Kim wipe and then quadruple rinsed it with distilled water. I then rinsed it four times in a

solution of acetone:methanol (2:1 v:v). I used acetone in this step instead of the dichloromethane used in all other steps of the procedure, because of safety concerns with the lack of fume hood and proper facilities at the Ephorate. After removing the outer skin of the sherd and discarding it, I typically ground off 2.0-4.0 g of each sherd and collected the fine pottery dust in the baked aluminum foil, which I then funneled into a pre-weighed, solvent-washed vial. The only exception to this amount collected was with the fine wares that were too thin to warrant a larger sample. I collected less than 1.0 g from seven fine ware sherds with a range 0.2-0.9 g in weight. For every seven samples collected, I created one blank. In total, I collected 115 samples and 17 blanks. Upon returning with the samples to the United States, I resumed chemical processing of the samples at the Archaeological Research Laboratory (ARL) at the University of Tennessee-Knoxville. The samples were stored in a refrigerator until ready to be chemically processed.

Lipid Extraction Protocol

At the ARL, I rinsed all glassware in a solution of ultrapure water and Contrex glass cleaner for 24 hours, scrubbed each piece four times, and rinsed each 10 times in ultrapure water. I rinsed items that could not be baked in the furnace (volumetric flasks, and graduated cylinders) 15 times in ultrapure water, left them to dry in a secondary fume hood, and rinsed them with dichloromethane and methanol 25 times each prior to use. Additionally, I rinsed plastic/Teflon caps 10 times in ultrapure water, soaked them in hexane for 10 minutes, and left them to dry prior to use. I baked all glassware with the same procedure outlined above.

The overall extraction method employed in this study was first established by Evershed et al. (1990). The exact protocol that I followed was developed by Dr. Eleanora Reber at the University of North Carolina –Wilmington Pottery Residue Laboratory (Reber 2014).

I added an internal standard of *n*-Tetratriacontane (Ultra Scientific) in hexane (Optima; Fisher Chemical) in the amount of 10 µl or 20 µl per sample, depending on sample size. Samples under 3.0 g were given 10 µl; samples 3.1 g or larger were given 20 µl. Then, I added 10 ml of 2:1 dichloromethane/methanol (v:v; Optima, Fisher Chemical Company; Ultra Resi-Analyzed, JT Baker [respectively]) to each sample. Henceforth, I will refer to this solvent as 2:1 solvent. I ultrasonicated the samples for two rounds of 20 minutes with a 10-minute intermittent rest. I transferred the samples to baked, solvent-cleaned glass centrifuge tubes with solvent-washed aluminum foil caps. I centrifuged the samples for 20 minutes at 2,000 rpm to separate pottery particles from the extracted lipids in liquid. I pipetted off the supernatant into solvent-washed vials and evaporated the liquid to dryness with nitrogen gas and mild heat.

The very fine size of the pottery particles warranted additional filtering with glass filter paper (GF/A size), which I baked prior to use at 400º C for four hours. I rinsed the filter paper, funnel, and vial four times with 2:1 solvent prior to filtering each sample. I reconstituted the dry sample in 2:1 solvent. I dumped the solvent into the glass funnel and refilled it for four cycles to maximize recovery of lipids from the master sample. I finished with an additional pipette full (approximately 2 ml) of straight 2:1 solvent poured into the funnel paper. This vial was then blown down with nitrogen, labeled as the master sample, and refrigerated.

I subsequently redissolved the master residue sample in 2:1 solvent for fractionation into three components to be analyzed—total lipid extract (TLE), fatty acid, and neutral. I transferred an aliquot (1/10 volume; 2 drops) to a solvent-washed glass vial with a fused glass insert for the TLE and another aliquot (9 drops) to a solvent-washed culture tube with PTFE lined cap. Both were blown down and the TLE was stored for later analysis.

The next phase was the creation of the fatty acid and neutral portions. I added 1-2 ml of sodium hydroxide (ACS Reagent grade; Ricca Chemical Company) in methanol to each aliquot in the culture tubes. I added an additional 0.5-1.0 ml to the samples that were darkest in color. I stirred each culture tube well and placed them in a hot water bath for three rounds of 20 minutes at 70-80° C. Between rounds, I removed each culture tube and stirred it. After each culture tube cooled, I extracted the neutral fraction by adding 2 ml of hexane, stirred the sample, and pipetted off the top layer into a new hexane-washed vial. This process was repeated three times, whereby the neutral extraction was complete. Then, I acidified the samples with drops of 2M hydrochloric acid in ultrapure water solution. I added enough drops to bring the pH down to 3-4. Occasionally, the samples became too acidic because of the difficulty in controlling drop size from the disposable pipette. I recorded the final pH of all samples.

Finally, I extracted the fatty acids with the same hexane method as I used for the neutral extractions. I blew down both fatty acid and neutral fractions to dryness. I later reconstituted these fractions with 10 drops of 2:1 solvent and split them into smaller samples to be injected into the GC/MS machine. I pipetted about 5 drops, half of the total amount, into solvent-washed, labeled 2 ml vials and then blew them to dryness.

Within 24 hours prior to injection, I derivatized all samples to be run with BSTFA (N,O-Bis(trimethylsilyl)trifluoroacetamide) with 1% TMCS (trimethylchlorosilane) (prepared mixture from Sigma-Aldrich). Derivatization with BSTFA changes the structure of the molecule by attaching non-polar trimethylsilyl units to the reactive functional group of the molecule, thereby making it more volatile and less polar (Barnard, Dooley, and Faull 2007:47; Brown and Brown 2011:63). The TMS esters, as they are referred to, are less likely to be reactive to other molecules or themselves (Brown and Brown 2011). I stirred in two to four drops of BSTFA per sample and then heated the samples at 60-70° C for 40 minutes. I evaporated the samples down with nitrogen for a final time and then re-dissolved them in two drops of hexane for GC injection.

Analysis and Interpretation

I analyzed samples on an HP 6890 GC coupled with a 5973 MS with diffusion pump using a 15 m DB-HT1 column with a diameter of 0.32 μm and a film thickness of 0.10 μm. Prior to the daily runs, I tuned the GC/MS with per-fluoro tributylamine (PFTBA) and monitored air, water, and carbon dioxide levels to check for leaks in the system. The carrier gas was ultrahigh-purity helium (Airgas). Head pressure was maintained at 0.5 psi at 50°C and the inlet temperature was 250°C. A constant flow of 2.5 ml/min was set during the run. Masses were scanned within the range of 40-800 m/z. All samples were injected with a volume of 1μL into the GC/MS instrument in splitless mode. If the sample was overloaded with compounds, as evidenced by the lack of compound separation in the gas chromatogram, the sample was reinjected with a volume of 0.2 μL to decrease the concentration. For total lipid and neutral extracts, the

temperature was held for 2 minutes at 50°C, ramped at a rate of 10°C/minute until 350°C was reached, and then held for 10 minutes for a total run time of 42 minutes. The temperature program for the fatty acid extracts started with a 2-minute hold at 50°C. It was then ramped at rate of 15°C/minute until 150°C was reached, ramped at rate of 3°C/minute until 250°C was reached, and then held for 10 minutes for a total run time of 52 minutes. Data acquisition was performed with Chemstation software with NIST database.

This lengthy procedure of extraction and instrumentation produces three gas chromatograms with combined mass spectral data for each sample—total lipid extract (TLE), neutral (N), and fatty acid (FA) fractions. An example of one of the TLE chromatograms is shown (Figure 3.1). Each of these fractions can reveal different compounds in the mixture. The TLE is an overall record of compounds recovered from the sample, including many of the compounds found in the neural and fatty acid components. Found only in the TLE are triacylglycerides (TAGs), diacylglycerides (DAGs), and monacylglycerides (MAGs), as well as wax esters. Since the TLE is extracted and run prior to saponification, which breaks ester bonds and releases the fatty acids, hydrocarbons, and alkanols, these larger compounds remain intact. The fatty acid portion includes original and recently freed fatty acids along with occasional MAGs and DAGs that did not fully saponify. Broad distinctions between plant, marine animal, microbial, and terrestrial animal can be drawn from relative proportions of fatty acids (Evershed 1993:85). The neutral component will be everything else, including free glycerol molecules, alcohols, sterols, hydrocarbons, etc.

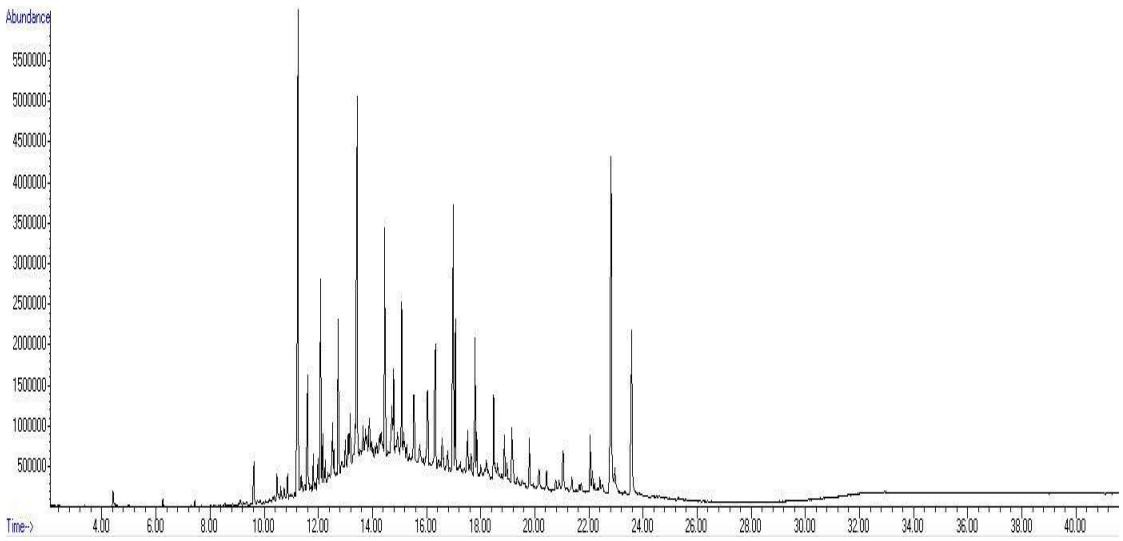

Figure 3.1 Gas chromatogram of the TLE of sample 2062

The approach to understanding residues is to identify the compounds, compare compound ratios, identify any biomarkers present, and interpret the entire distribution of compounds across all three components (Evershed 2008b:27). Biomarkers can be present in any of the three components. Fatty acids are ubiquitous and do not tend to be as diagnostic as singular compounds. One exception to this is branched $C_{15:0}$ and $C_{17:0}$ fatty acids, which are microbial biomarkers (Evershed 1993). Biomarkers are most often found in the neutral component. Sterols and terpenoids in particular tend to be very diagnostic compounds. However, they are generally present in smaller quantities than fatty acids in lipid extracts (Evershed 1993:80). Examples of biomarker sterols that differentiate plant from animal are cholesterol, campesterol, and β-sitosterol. Cholesterol is a biomarker for animals where it is found in higher proportions, while campesterol and β-sitosterol are only found in plants (Evershed 1993). Biomarkers can also be a suite of compounds, as is the case with beeswax or certain leafy greens. Evershed pointed out that all compounds in a biomarker suite must be present to attribute a residue to certain resource; otherwise the identification could reasonably be something else (Evershed 2008a). Complicating this further, the biomarkers produced by bacteria can show up in the lipid profile as well (Eglinton and Logan 1991:322).

A NIST database search is a starting point in compound identifications. However, the search regularly returns only low probability or incorrect results due to the degraded nature of the archaeological residue and the lack of archaeologically specific compounds in the database. Commercially available databases, as this one, have been compiled mostly from economically and agriculturally important plants and animals of today, not from what may have been used in the past, which is likely to be more extensive

(Evershed 1993:79). To manually identify compounds, I compared mass spectra published in other sources, such as peer-reviewed journals, US government agricultural databases, etc. or identified them based on known fragmentation patterns of specific compound classes. For the latter, I used Murphy (1993) for the analysis of mass spectra. I quantified lipids by comparing the peak areas in the TLE to the amount of known internal standard that was added per sample. Any sherd with less than 5µg/g of lipid can be considered functionally empty (Evershed 2008b:28; Reber et al. 2018).

I randomly selected a representative group from the 115 pottery samples that were originally collected for this phase of the project, as time constraints did not allow for all to be analyzed. I implemented stratified subsampling, whereby I separated the samples by pottery type (coarse, medium, and fine ware) and stratigraphic level, in order to ensure that all levels and fabrics were represented. I selected all samples from categories that had three or less and selected roughly half of the samples from the categories that had four or more samples for a total selection of 73 samples. The remaining 42 samples will be analyzed in a future phase of the project. From this point forward, any time I refer to the total group of samples, I am referring only to this subset.

With an understanding of lipids, the principles and historic trajectory of organic residue analysis, and the methodology employed in this study, I present a detailed analysis of the results from Ayia Triada samples in the next chapter.

CHAPTER FOUR
RESULTS

In this chapter, I discuss the results of organic residue analysis of the Ayia Triada pottery samples. First, I detail some initial contamination concerns that partially affected residue data. I then provide a data overview, including quantity of residue, level of preservation, and ubiquitous compounds. Next, I discuss the lipid distribution studies I conducted with the multi-area sampling of nine vessels. I follow with an in-depth discussion of the fine, medium-coarse, and coarse ware residue results. Subsequently, evidence for beeswax found in samples will be discussed. Finally, I further discuss a series of high-yield residues at the close of the chapter.

Contamination Concerns

As mentioned in Chapter 3, solvent blanks were created and subjected to the entire extraction process to serve as a control for contamination, which yielded three sets of blanks corresponding to the major fractions (TLE, N, FA). These can be used to pinpoint if and when contamination occurred in the extraction process. There are generally two ways to evaluate contamination levels in blanks. One way is to examine the relative abundance of all compounds across the blank; another is to look for the presence of biomarkers that could be interpretively important and therefore skew the results (Eleanora Reber, personal communication 2018). I decided to take a somewhat combined approach: I assessed the pervasiveness of any biomarker(s) found in the blank and its associated samples and determined if there was a large relative abundance of other, more

ubiquitous compounds to be problematic. I calculated the amount of biomarker relative to the internal standard of the blank. If the amount of the biomarker in the blank was greater than 1% of the internal standard, it was flagged for further consideration as described below.

Overall, the TLE blanks looked 'clean', meaning that they were free from significant contamination. Two of the 16 TLE blanks were not as 'clean' overall as the others, Blanks 9 and 14, suggesting that they may have been contaminated at some point in the extraction process. Blank 9 contained a relatively large amount of an interpretively important biomarker, β-sitosterol. It was present in 1.16% relative to the amount of internal standard. All samples associated with this blank, 7063, 5665, 10335, 9607, and 5486, were examined further for contamination. Two of the samples, 7063 and 5665, did not have β-sitosterol in them and therefore were not discarded. Samples 10335 and 9607 did have β-sitosterol. The amount present in the blank was between 30% and 77% of the amount found in these samples. A cutoff of 5% of the blank compound relative to the associated sample was the working limit for biomarkers I adopted in this study, such as sterols and diterpenoids. Since the blank contamination was contributing such a large amount of β-sitosterol to the sample, these samples were discarded. It was more difficult to parse out whether the residue in the last sample, 5486, was purely the result of contamination. It contained β-sitosterol, but it was present in significantly larger quantities in the sample than in the blank. The blank amount of β-sitosterol was only 2% of the amount in the sample. The peak area of β-sitosterol in the blank was subtracted from its area in 5486, and the sample was kept as part of the analysis.

The other possibly compromised blank, Blank 14, had significant contamination in the form of fatty acids and some β-sitosterol. The β-sitosterol was in a low enough abundance at 0.38% of the internal standard to not discard the associated samples completely, but the range of fatty acids present in the blank was concerning. Blank 14 had myristic acid ($C_{14:0}$), two isomers of $C_{16:1}$, linoleic acid ($C_{18:2}$), two isomers of $C_{18:1}$, and arachidic acid ($C_{20:0}$) along with the more ubiquitous $C_{16:0}$ and $C_{18:0}$ and their MAGs. These compounds were also present in the associated samples, 7055, 5690, and 5007, in substantial amounts relative to the amount in Blank 14. These samples appeared to be contaminated and were discarded from further analysis.

As a side note, Blanks 1, 5, 13 also contained biomarkers, namely β-sitosterol, Δ^5 avenasterol, and/or dehydroabietic acid (DHA), but they were present in amounts <1% relative to the internal standard in the blank. The amounts were low enough that they could be safely subtracted from the sample amounts and would not compromise the full set. The presence of any of these biomarkers, however, will be factored into the overall interpretation of the associated samples. These biomarkers will only be interpreted if there is a significant amount found in the sample to override the effect of the blank contamination, as in sample 5486 above. The source of these biomarkers in the blanks is unclear. The sterols β-sitosterol and Δ^5 avenasterol could originate from the culture tube caps used in the extraction process (Eleanora Reber, personal communication 2018). All of the TLE blanks contained small amounts of $C_{18:0}$ and $C_{16:0}$ and their MAGs. The area of these compounds found in the blanks were subtracted out from the corresponding area in the associated samples. Although useful, these compounds are not biomarkers and are ubiquitous compounds in nature.

The second set of blanks, the neutral blanks, overall looked acceptable, except Blanks 1, 5, and 9. Most of the neutral blanks contained small amounts of three alkanes, n-docosane, n-tricosane, and n-tetracosane. These were subtracted from the amounts in the corresponding samples, as the fatty acids (FA) and MAGs were treated in the TLE blanks. Blank 1 had β-sitosterol at 6% of the internal standard, which is considerably above the 1% threshold. The neutral portions of the samples associated with Blank 1 were excluded from analysis (5217, 653, 5175, 1686, 8428, 15787). Blank 5 was considerably better, but DHA was present 1.09% and β-sitosterol at 0.76% of the internal standard. Again, the biggest concern is the compound above the 1% threshold, DHA in this case. Samples associated with this run were 7489, 5905, 14750, 3133 A, and 1656. Samples 7489 and 5905 had no DHA and therefore were not discarded. Samples 14750, 3133 A, and 1656 had DHA, but in far greater amounts than in the blank. The DHA amount in the blank was subtracted from the samples. However, the presence of DHA in this neutral blank will be taken into consideration with the residue interpretation of these samples. I will only interpret DHA in samples if they contain a large amount of it in the sample. Blank 9 had β-sitosterol at 1.49% relative to the internal standard in the blank. Its associated samples were 5486, 7063, and 5665. Sample 7063 had no β-sitosterol, while sample 5665 only had a small amount of it. This compound will not be interpreted in the latter sample. Sample 5486 had a sizable amount of β-sitosterol to which the blank β-sitosterol was only contributing 4%. The area of β-sitosterol from the blank was subtracted out.

Unfortunately, the fatty acid blanks did not fare as well. A wide range of fatty acids was recovered in these blanks. It is apparent that significant contamination occurred

in this last extraction step. Possibly, it was caused by cross contamination from the lab bench surface onto the pH paper used in the acidification step. It also could have stemmed from the culture tube caps, which are reusable but cannot be baked in the furnace, or even from the water used to make the HCL solution (Eleanora Reber, personal communication 2017). The latter is less likely, because ultrapure water was used at all stages. Regardless of its origin, this level of contamination is nearly impossible to tease out from the samples that accompany each blank. The fatty acid fraction for all samples was discarded. Fortunately, since the entrance point for the contamination is localized at the last major step of the extraction process, there is no evidence to suggest that the TLE and neutral fractions are compromised. Unfortunately, the information that this fraction could have provided is lost. To that end, FA ratios cannot be calculated. Any fatty acids that are reported throughout this chapter and the remaining ones are those that were recovered from the TLE portion. This is not ideal, but the presence/absence of certain fatty acids in the TLE can still provide valuable information.

Another area of concern is modern contaminants, which have a tendency to creep into chemical residue analyses in some form no matter how many precautions are taken (Evershed 1993; Heron and Evershed 1993; Mazow et al. 2014). This study is no exception. The following compounds were identified as modern contaminants. Although not contamination per se, polysiloxanes were found in nearly all residue results. These compounds are common breakdown products from the chromatography column. A common class of modern contaminants that were found is phthalates, which are compounds found in plastic. They are stable, frustratingly pervasive compounds in our modern environment and are easily recognizable via their mass spectra of peaks at m/z

149 and m/z 167 (Oudemans and Boon 1991:223). Two common amides were found in the samples and blanks, octadecenamide and hexadecenamide, and likely derive from curation materials (Reber et al. 2015:40). Polysiloxanes, amides, and phthalates were removed from the results without further scrutiny.

A few other modern compounds are of concern, because their presence could indicate that residues were contaminated by lipid-rich sunscreen lotion or bug spray and could lead to false interpretation of the archaeological vessel contents. DEET was found in nine samples. It was present only in minor amounts that ranged from 0.02-0.18% of the residue. Because DEET was present in such small quantities, it was removed from the sample without further consideration. Sunscreen markers, specifically octinoxate and octocrylene, were found in many more samples than DEET was; in all, 58 samples contained one or both of these, which totals 78%. These samples required additional scrutiny to determine if the extracted residues derived from the fillers, fragrances and other compounds that are often present in sunscreen lotions instead of being archaeologically-derived. Two scenarios are suspect for sunscreen contamination: a residue that looks unusually complete with a large amount of TAGs; or a residue that has a high abundance of unsaturated fatty acids and appears almost marine-derived (Eleanora Reber, personal communication 2018). Marine-derived residues usually have polyunsaturated fatty acids, several monounsaturated fatty acids, and a large relative amount of unsaturated to saturated fatty acids (CoBabe and Pratt 1995; Guitart et al. 1999; Reber and Evershed 2006). I examined the samples with sunscreen biomarkers for either of these scenarios. None had any TAGs or enough unsaturated fatty acids to be suspect; no samples were thrown out for these reasons. However, three samples contained

a large relative abundance of sunscreen markers to still be suspect. I established a standard that any sample containing sunscreen markers above 5% of the total lipid content was too high; three samples fell into this category with between 5% and 13%—10425, 8132, and 13144. To be cautious, these were discarded and excluded from further analyses. The rest of the samples yielded 4% or less of sunscreen markers relative to the overall lipid content, the overwhelming majority of which were present in relative abundances of <1%. These do not give any indication that they are significantly contaminated. Several samples also contained traces of α-tocopherol, vanillin, and cedrol. Although these could reflect the original vessel contents, they could also be derived from modern personal care products (Reber 2017; Eleanora Reber, personal communication 2018). Since these were found in samples that also contained sunscreen biomarkers and/or DEET, it is likely that they too originate from bug spray or sunscreen solutions. These compounds were removed. In summary, eight samples were discarded due to possible contamination, either post excavation or laboratory contamination.

Data Overview

The full results are presented in the Appendix with each sample's quantity, stratigraphic location, pottery type, residue description, and interpretation. These results will be categorized by their fabric types and discussed in later sections of this chapter. Table 4.1 provides a condensed overview of the data set by fabric type and includes samples that were discarded, imported wares, and samples that belong to a pair.

Quantification

With the extent of contamination determined and suspect samples discarded, residues from the remaining 65 samples were quantified, as outlined in the Methods chapter. Prior to quantifying, I subtracted out of the all modern contaminants discussed above from the samples. All compounds that remain, both unidentified and identified, are presumably archaeologically-derived. Several of the blank *n*-TTC peak areas were higher or lower than the peak area of *n*-TTC in the sample itself by an order of magnitude. For example, a certain blank had 300,000 peak area of *n*-TTC and the sample had 30,000 *n*-TTC. To prevent overestimating or underestimating the amount of contamination, I adjusted any sample-blank pair where there was an order of magnitude difference between them.

Of the 65 samples, 94% had extractable residues above the cited threshold of 5µg/g (Evershed 2008b; Reber et al. 2018). Only four samples, 7183, 7684, 7538, and 8156, yielded too little interpretable residue and were not analyzed further. On the other end of the spectrum, the highest quantities recovered were around 1,000 µg/g. Sample 3133 A had 1,001 µg/g and sample 6682 had 915 µg/g lipid content. The distribution of quantities across the samples is shown (Figure 4.1). A total of 21 samples had quantities of 100 µg/g or more, roughly a third of the total. Most of these samples were from medium coarse vessels, while only one was a fine ware. In terms of vessel type, most of these above 100 µg/g were storage jars. However, one must keep in mind that for the majority of sherds in this study, the vessel type is not known. An equal number of these high yield samples were from unknown vessel types.

The mean lipid quantity of all samples was 117.6 µg/g. The overall spread of the data was characterized by compound class, for example, alkanes, fatty acids, alkanols, ketones, etc., and the relative abundance of each compound class was calculated, as well as the quantity for each compound class. Table 4.2 shows mean lipid class values for all samples.

Since there was such a high recovery rate, it is useful to explore possible reasons that could explain the lack of appreciable residues in four samples. One sample with negligible residues was a fine ware (8156), another was a medium-coarse ware (7538), and the last two were coarse wares (7183 and 7684). The coarse ware and medium-coarse ware samples were on the larger end of the sample size spectrum, ranging from 3.0 g to 4.3 g, which makes the lack of residues in the sherds even more pronounced. Sample 7538 was a thickened and flaring neck that belonged to a sizable storage vessel with medium coarse fabric. The sample was taken where the neck meets the shoulder. A few possibilities arise for its lack of lipids: the vessel was holding a liquid that was not rich in lipids; the vessel was never filled to the neck; the vessel was holding dry goods; or the vessel was empty (Cramp, Evershed, and Eckardt 2012:105). It was found in Trench 9, Unit 1. The stratigraphic location yielded plenty of other vessels with appreciable residues. Unfortunately, there are no comparanda in this study, as sample 7538 was the only neck sampled.

Samples 7183 and 7684 were both coarse-ware bases, from which a large amount of sample was taken. In fact, 7183 was one of the largest samples collected at 4.3 g. It was a thick, raised ring base of unknown vessel type. Sample 7684 belonged to a thinner

Table 4.1 Coarse, medium coarse, and fine ware samples with their associated stratigraphic location and vessel type when known. Paired samples from the same vessel are marked with letter designations

	Samples	Trench	Level	Unit	Vessel Type	Pairs	Excluded	Imported
coarse	653	8	4	1				
	1686	4	4		Jar			x
	3035	8	4					
	3049	8	4					
	3133 A	8	4			e		
	3482	12	4		Open, Handled	c		
	4972	9	5a		Jar	f		
	5007	9	5a				x	
	5114	11	4					
	5217	9	5b					
	5436	9	4		Jar	h		x
	5448	9	4		Jar	i		
	5510	9		2	Bowl			
	5665	9		2				
	5920	9		3				
	6682	11	5a					
	7055	11a	5				x	
	7056	11a	5		Jar			
	7183	11	5b					
	7489	9		1		e		
	7608	9		1	Jar	i		
	7623	9		1				
	7684	9	4		Jar	h		x
	7760	9		1				
	8096	9	5a		Jar	f		
	8141	9		1				
	8506	9	4		Jar			
	8849	11	4					x
	11277	12	4		Jar	i		
	11309	8	4	1	Open, Handled	c		
medium coarse	1656	4	4					
	2991	8	4					
	3038	8	5b		Jar	b		
	3040	8	5b		Bowl	a		

Table 4.1 Continued

	Samples	Trench	Level	Unit	Vessel Type	Pairs	Excluded	Imported
medium coarse	3198	8	4					
	3275	8	4					
	3292	8	4		Jar	b		
	4469	9	5a		Jar	d		
	5025	9	5a		Jar			
	5112 A	11	4					
	5175	11	5b		Bowl			
	5278	9	5a					
	5475	9		2				
	5489	9		2				
	5905	9		3				
	6683	11	5a					
	7063	11a	5		Jar			x
	7064 B	11a	5					
	7064 A	11a	5					
	7184	11	5b					
	7538	9		1	Jar			
	7599	9		1	Bowl			
	8020	9		1	Jar	d		
	8072	9		2				
	8092	9		1	Bowl			
	8428	9	4		Bowl			
	10335	Balk B	4				x	
	10421	Balk A	4/5b					
	12461	Balk A	4					
	14750	Balk A	4					
	15787	8	3		Bowl	a		
fine	129	9	4		Pyxis			x
	2062	8	4		Sauceboat			
	5486	9		2	Sauceboat	g		
	5690	9	5a				x	
	7078	11a	5					x
	7219	11a		3b				x
	7370	9		1	Sauceboat	g		
	8132	9					x	

Table 4.1 Continued

	Samples	Trench	Level	Unit	Vessel Type	Pairs	Excluded	Imported
fine	8156	9		1				x
	9607	12	4				x	
	10425	Balk A	4/5b				x	
	13144	Balk B	4	1			x	

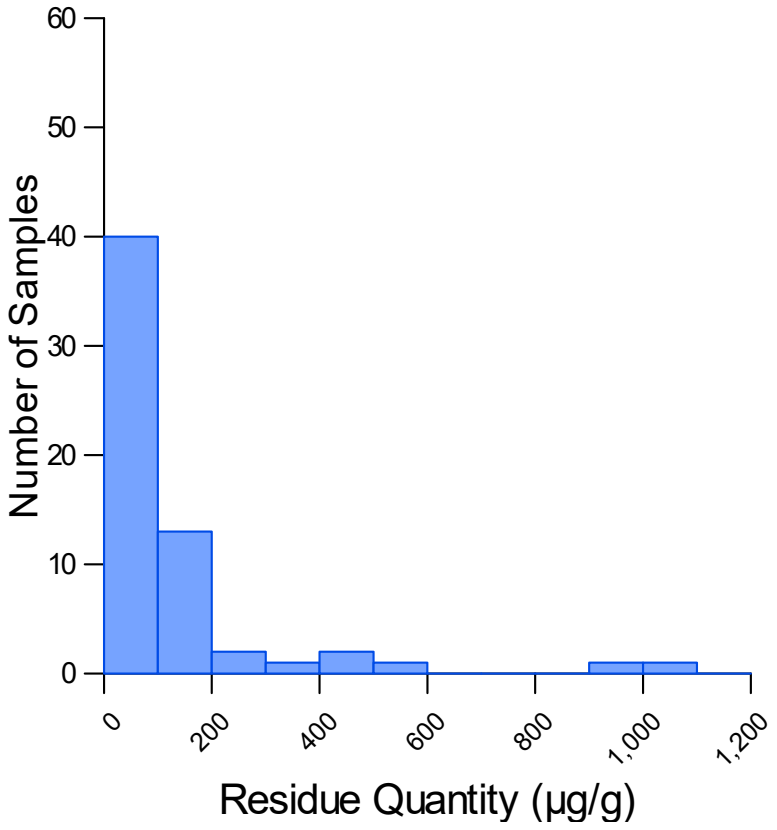

Figure 4.1 Histogram of Ayia Triada sample quantities

Table 4.2 Compound classes identified in Ayia Triada samples with mean, standard deviation, maximum value, and sample with the maximum value

Compounds	Mean (µg/g)	Standard Deviation	Max Value (µg/g)	Sample with Max Value
Alkane	27.3	+/- 54.3	378.3	3133 A
Alkanol	39.8	+/- 92.6	510.9	6682
Free fatty acid	19.2	+/- 37.4	263.2	7623
Sterol	1.5	+/- 2.4	12.4	12461
MAG	3.4	+/-3.6	14.9	5175
DAG	1.0	+/- 1.6	6.4	8096
Wax ester	6.0	+/- 10.0	36.4	6682
Ketone	4.3	+/- 10.5	32.3	6682
Diol	15.7	+/- 23.4	73.6	3133 A

walled vessel, specifically an ovoid, constricted neck jar. This jar was sampled also on the body, which will be discussed in the Lipid Distribution Studies section. Bases are the only part of the vessel that in necessarily in contact with the vessel contents, unlike the upper body or the necks which depend on the level of dry or liquid contents inside. Therefore, the fact that these bases had negligible residues suggests that either they were carrying resources with a low lipid content, such as certain liquids or dried goods, or that they were empty (Cramp, Evershed, and Eckardt 2012:105). Another possibility is that the application of heat caused a depletion of lipids. Lipids are less likely to survive in bases if the vessel was used on an open fire, because the intense heat at the base can destroy lipids (Charters et al. 1993). This seems unlikely for these samples, since there was no sooting or burning marks noticed on these sherds.

Sample 8156 was the only of the six fine wares with quantities below 5 µg/g. The vessel type to which this sample belongs is unknown. Its fine, grey fabric is coated with a darker black slip on the interior and exterior. Two other samples were made of the same fabric, but neither had this slip. A small amount of sample (1.2 g) was collected, because the sherd itself was rather small. It is possible that the slip prevented the absorption of lipids into the vessel walls. This vessel could have been a small pyxis or a small bowl based on its size and curvature, but it is too small to be sure.

Preservation

The level of preservation is partly indicated by the residue quantity, but also by the type of compounds that are preserved. Excellent preservation is marked by the presence of TAGs, for instance. These are large molecules with three esterified FA that

are highly susceptible to degradation (Scrimgeour and Harwood 2007). TAGs were not identified in any of the samples. However, DAGs were identified in most of the samples (57%) and MAGs were identified in all. The conversion from acylglycerides into free fatty acids is not complete in these samples, which indicates a moderate level of preservation.

Certain other lipids are easily carried off by groundwater and indicate a good level of preservation if found in the TLE. They tend to be lower molecular weight compounds, such as short-chain dicarboxylic acids, or diacids, with up to 12 carbons, short-chain FA, and glycerol (Copely et al. 2005:865; Regert et al. 1998:2029). The diacids are usually found only in the FA portion of the residue, because the free diacids in the TLE are easily washed away and the bound diacids require base treatment to extract (Regert et al. 1998:2029). Their presence in the TLE, therefore, suggests minimal action from leaching and good preservation of the lipids. A total of 21% contained short-chain α-ω dicarboxylic acids in the TLE. Shorter chain FAs, up to $C_{10:0}$, were found in 46% of samples in the TLE. The presence of glycerol in the TLE is also a good indicator of preservation, because glycerol is easily washed away in groundwater (Copely et al. 2005:865). Seven of the samples contained glycerol in the TLEs, most of which correspond to the samples with high residue quantities. Nearly 51% of the samples preserved low molecular weight compounds, either glycerol, dicarboxylic acids, short chain FA, or a combination of these compounds, underscoring a good level of preservation overall.

Common Compounds

Labdane

One compound stands out for its ubiquity within the samples. It has a strong peak at m/z 257 and a minor one at m/z 272 (Figure 4.2). It elutes close to palmitic acid ($C_{16:0}$). It is possibly some type of labdane diterpenoid, which would indicate a plant resin (Eleanora Reber, personal communication 2017). It appeared in 97% of the sample TLEs, including the four samples that had < 5 µg/g. It was present in relatively low quantities in most samples, except for 5175 where it was present in 10.3 µg/g. Sample 5175 belongs to a medium to large bowl. A few other samples contained labdane within the 2.0-4.2 µg/g range: 8020, 653, 2062, 3482, 3292, 1686, 5217, and 7623; the remaining samples had labdane quantities of <2.0 µg/g. This compound was also found in the fatty acid portions. Labdane quantity does not seem to correlate to the amount of residue preserved, the amount of sample collected, or with samples containing other resin biomarkers. The four highest yielding residues reflect how variable the labdane quantity is. In respective order for the highest quantity samples (3133 A, 6682, 6683, and 5175), labdane quantities were 0.7, 0.7, 1.1, and 10.3 µg/g. Furthermore, labdane cannot be attributed to laboratory contamination, as it was not found in any solvent blanks, nor was it found in either of the hexane blanks that were run prior to each day's GC/MS runs.

A compound being this widespread across fabrics, vessels, and sherd types is somewhat unusual. There are several possibilities for this. It could have been a substance added as a sealant to the pottery (Reber and Hart 2008) or it could have been an ingredient processed or consumed in all pottery. However, the fact that it is found in

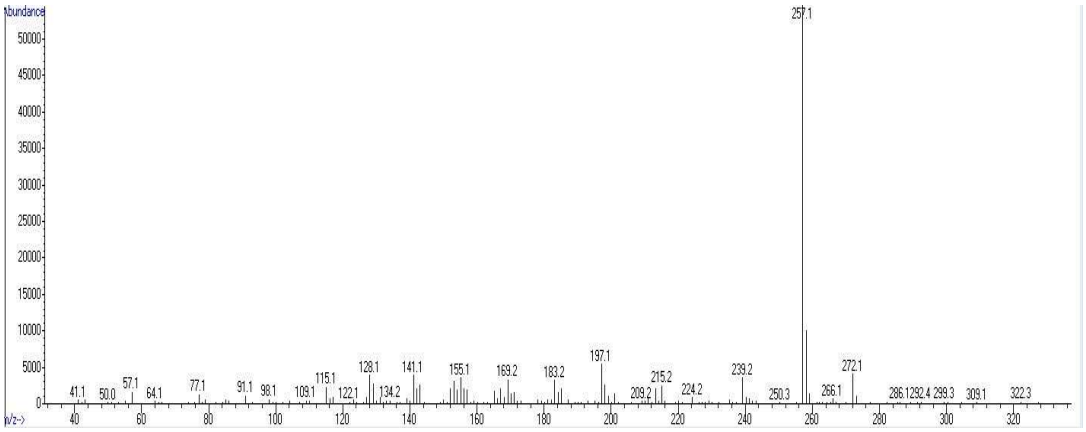

Figure 4.2 Mass spectrum of labdane-type compound

imported and local wares and equally in serving and storage vessels, which themselves seem to have a range of residues, seems to discredit this. Another possibility is that labdane absorption was the result of a post-depositional process. It could have been a substance poured over all pots or over the entire activity area, possibly related to the feasting or ritual activities. It also could have been a recurrent ritual that happened regularly or when a new individual was buried. Contemporaneity of the burials cannot be determined (Mavridis and Tankosić 2016a:224). Alternatively, labdane could have absorbed into the pottery from a layer of brush or wood that was laid over the area and burned. There is evidence for burning with the macrobotanical layer in the cave. A resinous material could have provided the fuel for that fire. However, one would expect polycyclic aromatic hydrocarbons (PAH) as well if this were the case (Harvey 1998). PAHs have not been identified in any of the samples.

Labdane could also signify post-excavation contamination. All pottery was washed after excavated, several years prior to lipid extraction. Labdane could have originated from a soap or cleaner that was used. One way to test this theory would be to sample pottery from other periods and areas in the cave. Another way would be to sample pottery from other sites that was washed at the storehouse around the same time. Initially, I suspected that it came from powdered disposable gloves, which I had to use initially because of glove availability in Greece. Many of the highest quantities of labdane were found in samples collected before I switched to powderless gloves about two weeks into sampling, but not all. There are still some high labdane quantities in samples collected when I was exclusively using powderless gloves. Until further sampling is conducted, or

the next phase of sample analysis is completed, the origin of this compound remains a mystery.

Long-Chain Alkanols

The long-chain alkanols found in residues can typically be attributed to the epicuticular wax of higher plants, specifically alkanols (OL) with 20-34 carbons (Bianchi 1995:185; Tulloch 1976:245). Plants usually have one to four main alkanols within this range (Bianchi 1995:185; Tulloch 1976:245). These alkanols are also found in insect waxes, such as beeswax (Tulloch 1971). However, alkanols less than 20 carbons in length were found in many of the samples in this study, ranging from 12-19 carbons. They cannot be linked to any of the main plant components or to bacterial alkanols (Kolattukudy 1976). Even more intriguing is that in 49 samples (80%), 1-octadecanol (C18) and to a lesser extent 1-hexadecanol (C16) were the most abundant within the entire alkanol sequence, including the plant epicuticular plant range.

It is possible that these long-chain alkanols represent some sort of contamination. The compound 1-octadecanol was indeed found in some of the TLE and N blanks. However, the amount of 1-octadecanol relative to the internal standard in these blanks was <0.6%. These were insignificant amounts and were subtracted out from the samples. It is possible that these compounds originate from the very fine-grained filter papers used in this study. The blanks too were filtered in the exact manner as the samples, which would explain how 1-octadecanol could show up in the blanks. However, the filter papers were baked in a furnace to destroy any residual lipids and were rinsed with solvent four times prior to being used in the extraction process. Furthermore, if filter papers were introducing contamination, one would expect quantities to be similar across the blanks

and the samples, which is not the case. Another culprit is possibly the Kim wipes used to clean the drill bit between sampling. However, since Kim wipes were only used on true samples, 1-octadecanol should not be present in any of the TLE and N blanks.

Possibly, a more likely culprit is yeast. Yeasts are known to produce long-chain alkanols, specifically 1-tetradecanol, 1-hexadecanol, and 1-octadecanol and traces of 1-dodecanol (White, Hammond, and Rose 1987: 2188). It seems feasible that if organic material was not washed off the surface of the vessels after use, especially if sugary substances were involved, naturally occurring yeasts could have acted on them. Yeast exists in nature on fruits, for example. Various yeast species could also have been living in the cave itself, which could possibly explain why such widespread yeast breakdown occurred.

Lipid Distribution Studies

Identifying the distribution of lipids across a vessel's surface can aid in determining the original function of a vessel (Charters et al. 1993; Heron and Evershed 1993). Multiple sampling from a single pot has been conducted only a few times in the literature: Charters and colleagues' (1993) Raunds study, Reber and colleagues' (2015) Angel Mounds study, Cramp's (2008) dissertation, and Cramp, Evershed, and Eckardt's (2012) study. It is often impossible to sample from multiple locations on a single vessel, because one is rarely allowed to subject intact pots to destructive analysis. These studies instead used reconstructed vessels or those damaged *in situ*. A collection method that allows for multiple sampling of a vessel and prevents visible external damage is by shaving off layers as I did in this study, instead of removing and then crushing a chunk of

the pot sherd. Four sets of samples from different areas of the pots were sampled in this manner, as well as five sets of body sherds thought to belong to the same vessels (Table 4.3). The latter sets were collected in part to gauge to variability across the interior surface.

Sample Pairs from Different Areas

15787 and 3040

Sample pair 15787 and 3040 belonged to a medium-coarse bowl with an inverted rim. This set showed the greatest difference in quantity between different areas of the pot. Sample 15787 was collected from the rim and 13.6 µg/g of lipid was extracted, while 3040 was collected on the body closer to the base and 114.3 µg/g was extracted. Similar amounts of sherd were collected: 1.7 g for 15787 and 1.4 g for 3040; therefore, the observed difference in residue quantity cannot be attributed to sample size. Although 15787 had a smaller amount of residue, it generally agreed in content with 3040. Both contained cholesterol and the same tight range of alkanols, which indicates epicuticular wax from a limited range of plants (Tulloch 1976). The fatty acid range in both is narrow with $C_{14:0}$, $C_{16:0}$, $C_{18:0}$ and $C_{18:1}$. Sample 3040 has some $C_{22:0}$. Surprisingly given the significant difference in total lipids, the quantity of fatty acids is similar for samples 15787 and 3040 (6 µg/g and 9 µg/g). It is unusual how little free fatty acids were recovered from 3040, the higher quantity sample, especially considering the lack of DAGs and TAGs which should indicate acylglyceride breakdown. Unsaturation levels

Table 4.3 Pairs of sherds sampled from reconstructed vessels

Pair from the Same Vessel	**Rim**	**Body**	**Base**	**Vessel Type**
15787 and 3040	15787 *13 µg/g*	3040 *114 µg/g*		Bowl with inverted rim
3038 and 3292		Both 207, 165 *µg/g* (respectively)		Short and squat Jar
3482 and 11309		3482 *150 µg/g*	11309 *96 µg/g*	Open vessel with handle and flaring rim
8020 and 4469		Both *164, 119 µg/g*		Tall, bulbous jar
7489 and 3133 A		Both *16, 1001 µg/g*		unknown
4972 and 8096		Both *111, 106 µg/g*		Short and squat Jar
5486 and 7370		5486 *19 µg/g*	7370 *7 µg/g*	Urfirnis sauceboat
7684 and 5436		5436 *9 µg/g*	7684 *4 µg/g (empty)*	Ovoid jar
7608, 11277, and 5448		All *26, 47, 6 µg/g*		Jar

were very low for both. $C_{18:1}$ could derive from plants or animals (Scrimgeour and Harwood 2007:4). The lipid profile of both suggests a mixture of animals and a limited range of plant products.

The major differences between the sherds in this set were within the alkanes, sterols, and degree of degradation. Sample 15787 contained only alkane C20, while 3040 had the full range of alkanes from C18-33. Alkanes made up 51% of the TLE for sample 3040. Sample 3040 also contained a simple distribution that peaked at C24. This pattern is not usually attributed to plants, because plants have primarily odd-chain alkanes and minor amounts of even-chain alkanes (Tulloch 1976:243). This pattern could have arisen from burning plant material above 400º C, as Wiesenberg and colleagues (2009) have shown. However, burning would leave other biomarkers that were not found in this sample. The interior did not any show signs of burning either. It also could signify synthetic wax or fossil wax contamination (Evershed 2008a:899; Reber 2017:11; Regert et al. 2005). However, the alkane sequence does not peak in the right place for paraffin wax at alkane 27 or have a bimodal distribution like ozokerite (Regert et al. 2005:130). Likely this distribution is the result of microbial breakdown (Eleanora Reber, personal communication 2018). Sample 3040 also contained β-sitosterol, which was present in lower abundance than cholesterol. This suggests that another plant component was present beside plant wax. Microbial degradation is indicated in the body sherd by many branched alkanes and some branched alkanols. The rim sherd, 15787, showed far less degradation with only a small amount of $C_{15:0}$ suggestive of any degradation at all. It is worth noting that the neutral fraction of sample 15787 had to be discarded because of the blank contamination discussed earlier. It is likely that there were more sterols, alkanes,

and alkanols in the neutral fraction that would have been released during saponification, which could certainly account for this difference.

The lipid distribution of this pot with less at the rim and more towards the base does not support any interpretation that this vessel was a cooking dish. Heat would likely deplete or destroy the lipids at the base and lower body (Charters et al. 1993:216). The spatial patterning supports the interpretation of this vessel as a serving dish. One would expect more lipids in the belly of the bowl than towards the rim. This was confirmed by Charters and colleagues (1993), even though their overall quantities were far lower and the spread was less extreme than between 15787 and 3040. The body sherd also contained more diverse lipids than the rim, which again fits for a bowl, where only bits of the foods would make their way to the rim. However, the lipids that would be most likely to absorb anywhere would be the fatty acids, the more viscous compounds. This could explain the similar amount of FA extracted from the rim and the body. The degradation pattern, however, is different than has been observed elsewhere. It is thought that areas closer to the rim would have more degradation, because of the exposure to air, but the pattern is reversed in this vessel (Reber et al. 2015:45).

7684 and 5436

The sample pair 7684 and 5436 had little residue overall. In fact, sample 7684, a base, was one of the four samples with negligible residues. Sample 5436, a body sherd, did not contain much above the functionally empty threshold at 9 μg/g. Caution should be exercised here, because of the difference in sample sizes. Sample 7486 weighed 3.1 g, while sample 5436 weighed 4.1 g. Body sherd 5436 displayed a range of unsaturated FA, which is unexpected given its low quantity of residue. It had $C_{18:2}$ and $C_{16:1}$, which

suggests plant or fish components to the residue. Without the marine biomarkers of isoprenoid FAs, ω-(o-alkylphenyl)alkanoic acids, and $C_{17:1}$ and $C_{19:1}$, it cannot be definitively assigned to a marine source (Baeten et al. 2013; Hansel et al. 2004). It also contained $C_{18:1}$ and its the degradation product of $C_{9:0}$. $C_{18:1}$ could derive from plants, such as olive oil, or animals (Scrimgeour and Harwood 2007:4). If it were degraded olive oil, unsaturated TAGs, high quantities of $C_{18:1}$, $C_{9:0}$, and short-chain diacids would be expected (Gunstone and Harwood 2007; Mills and White 1994; Regert et al. 1998). The quantities of both $C_{18:1}$ and $C_{9:0}$ were unfortunately small. Cholesterol and an oxidized cholesterol derivative indicate animal products. One of the main phytosterols of higher plants, β-sitosterol, was found, in addition to a slightly less common one, Δ^5 avenasterol. The residue was microbially degraded, as evidenced by $C_{15:0}$, $C_{17:0}$, and branched alkanes and alkanols. There was an almost complete hydrolysis of acylglycerides, since there were no TAGs and free fatty acids were more abundant than DAGs and MAGs. A higher plant component was suggested by the alkane distribution dominated by alkane 31 and 29, in that order. The alkanol distribution was even-dominated from C20-28, which indicates that more than one plant was present.

The shape of this large, ovoid jar with a constricted neck suggests that it held a liquid of some sort. The lack of sooting and the low lipid amounts support this typological assessment. Other researchers have observed this pattern in jars. Charters and colleagues (1993) found smaller amounts of lipids in the bases of jars, but the spread between base and body was wider than in this case. They generally sampled the body at the midway point between the rim and base. The body sherd in this set could have come from a location closer to the base and therefore, more closely mimic base lipid

absorption. This distribution is most like that found in a Roman storage vessel with low lipids at the base and body, which the authors indicated could reflect dry or lipid-poor substances being stored (Cramp, Evershed, and Eckardt 2012:105).

11309 and 3482

Samples 11309 and 3482 were from an open, handled vessel. Similar amounts of sample were taken: 2.6 g from 11309 and 3.0 g from 3482. Both had evidence for a mixture of more than one plant and animal products with microbial degradation. Microbial degradation is suggested in both sherds by $C_{15:0}$, straight chain and branched $C_{17:0}$, $C_{19:0}$, branched alkanes, and branched alkanols. Sample 3482 had more branched alkanols and alkanes. The alkane sequence peaked at 20 in the neutral fraction of sample 3482 and at 24 in sample 11309. The alkane sequence in both followed the pattern of bacterial degradation. The alkanol sequence was only slightly wider in sample 3482 with the addition of OL 32. However, sample 3482 contained a more complex mixture overall. It had evidence for pine resin with 15-hydroxy-7-oxo-dehydroabietic acid, which is an oxidized form of dehydroabietic acid (Buckley et al. 2004:296). It contained wax esters with $C_{14:0}$, $C_{16:0}$, and $C_{16:1}$ fatty acid moieties. Furthermore, it contained ketones with 27, 31, 33, and 35 carbons, which suggests the cooking or heating of fatty substances in excess of 300° C in clay containers (Evershed et al. 1995; Evershed et al. 2002; Raven et al. 1997). The ketone with 33 carbons was the most abundant in both the TLE and the N fraction. Sample 11309 had one unique compound not found in 3482, laevulic acid.

Sample 11309 was a base/body and sample 3482 was a body sherd that appears to be lower on the body. It is difficult to determine how far apart these sherds would have been, since these pieces do not mend and the vessel is too incomplete to gauge its original

depth. The distribution of residue with less residue at the base and more further up on the body suggests the cooking of substances. The lower amount of lipids (although not drastically lower) would be caused by thermal degradation at the base. This distribution of ketones on the body and not on the base has been observed by other authors. However, the formation of ketones and oxidized cholesterol at the low to mid body could suggest the highest heat was there and not on the base (Cramp, Evershed, and Eckardt 2012:105). The ketone location could also reflect boiling of foodstuffs, because the liquid level of foodstuffs would maintain the highest temperature (Cramp, Evershed, and Eckardt 2012:105). Below that water would be evaporating out. Therefore, the temperature below that line would not be high enough to form ketones and would likely not have as many lipids. However, since a third sample at the upper body or rim was not taken, this cooking practice needs more evidence. This was the only vessel like this tested, so it is challenging to extrapolate further. Regardless, the lipid distribution and presence of ketones suggests that this was a cooking vessel.

5486 and 7370

Samples 5486 and 7370 likely belong to an Urfirnis sauceboat (Fanis Mavridis, personal communication 2018; Žarko Tankosić, personal communication 2018). They both have unremarkable range and quantity of fatty acids and MAGs. These sherds contained a strong plant component and a narrower range of plants than many of the other samples in this study. Sample 5486 had an unusually tight range of alkanes of 20, 21, 22, 24 with very little amounts of them. Plant-derived alkanols were limited with C20, 22, and 24 as well. Sample 7370 too had a narrow range of alkanes with only 22-25 and alkanols with 20 and 22. This could indicate that only one or two plants were present.

Sample 7370 contained minute quantities of cholesterol and β-sitosterol. However sample 5486 contained an unusual quantity and range of sterols (Figure 4.3). Sterols in this sample totaled 6.9 μg/g, which is over a third of its TLE residue. The three main plant sterols of higher plants were found: β-sitosterol, stigmasterol, and campesterol (Grunwald 1975:210). Of these, β-sitosterol and stigmasterol were present in significant amounts. Also, Δ^5 avenasterol, was found, which is related to sitosterol (Belitz and Grosch 1999:220). This plant sterol is found in many oils, such as rapeseed oil, olive oil, flax seed oil, and palm oil, as well as other plants, such as white mustard, walnut, oats, and lettuce, to name a few (Baocheng et al. 2014:6860; Belitz and Grosch 1999:220; Duke 1992; Kochhar 2002:318). It must be noted that Δ^5 avenasterol and β-sitosterol were found in the associated blank. They were present in a low relative amount to that found in the sample and as outlined earlier in this chapter, were subtracted out from the sample amount. Another compound was identified as poriferasta-7,25-dienol, found in nuts and seeds (Phillips et al. 2005:9440). Two unusual sterols were also found: a compound that is cycloeucalenol, cycloartenol, or oleana-11,13(18) diene; and a compound that is either cyclolaudenol or 24-methylene-cycloartenol. The chemical structures of these three compounds in the first case and two compounds in the second case are similar, which precludes differentiation on mass spectra alone. Regarding the former compound that could be cycloeucalenol, cycloartenol, or oleana-11,13(18) diene, comparison to known elution order suggests that this compound is actually the coelute of cycloartenol and cycloeucalenol (Kornfeldt and Croon 1981:307). A minor amount of cholesterol was found in this sample. Given that cholesterol is found in typically minor

amounts in plants and this residue is overwhelmingly plant-based, the cholesterol could be plant-derived (Behrman and Gopalan 2005; deMan 1999:51).

The three most diagnostic sterols—poriferasta-7,25-dienol, cycloeucalenol/cycloartenol, and cyclolaudenol or 24-methylene-cycloartenol—are found separately in a handful of commodities. However, the suite of poriferasta-7,25-dienol, cycloeucalenol/cycloartenol, and 24-methylene-cycloartenol is found in oils, specifically sesame seed, flax seed, poppy seed, and wheat germ oils (Jeong et al. 1975; Kornfeldt and Croon 1981; Phillips et al. 2005). Cycloeucalenol and 24-methylene-cycloartenol are also found in olive oil and rapeseed oil (Kornfeldt and Croon 1981). It is possible that poriferasta-7,25-dienol too is present in olive and rapeseed oil, but has not been identified yet. The researchers who reported poriferasta-7,25-dienol analyzed the oils of seeds and nuts but did not include rapeseed or olive oil (Phillips et al. 2005). Considering how well-researched both olive oil and rapeseed oils are, it seems unlikely that these would remain undetected in the scholarly literature. Cycloartenol and 24-methylene cycloartenol are found in gold of pleasure or camelina oil (Shukla et al. 2002:967). Shukla and colleagues (2002:967) reported several possible sterols that could not be identified from gold of pleasure oil. Poriferasta-7,25-dienol may be one of those unidentified compounds, which would make it another strong candidate for the identification of this residue. Although technically a smaller quantity is present in the base, the difference is not drastic.

The similar quantity between the base (7 µg/g) and body (19 µg/g) and lack of sooting suggests that this was not a cooking vessel. There was a substantial difference in sterol content of the samples. This could be explained by two factors. The Urfirnis slip

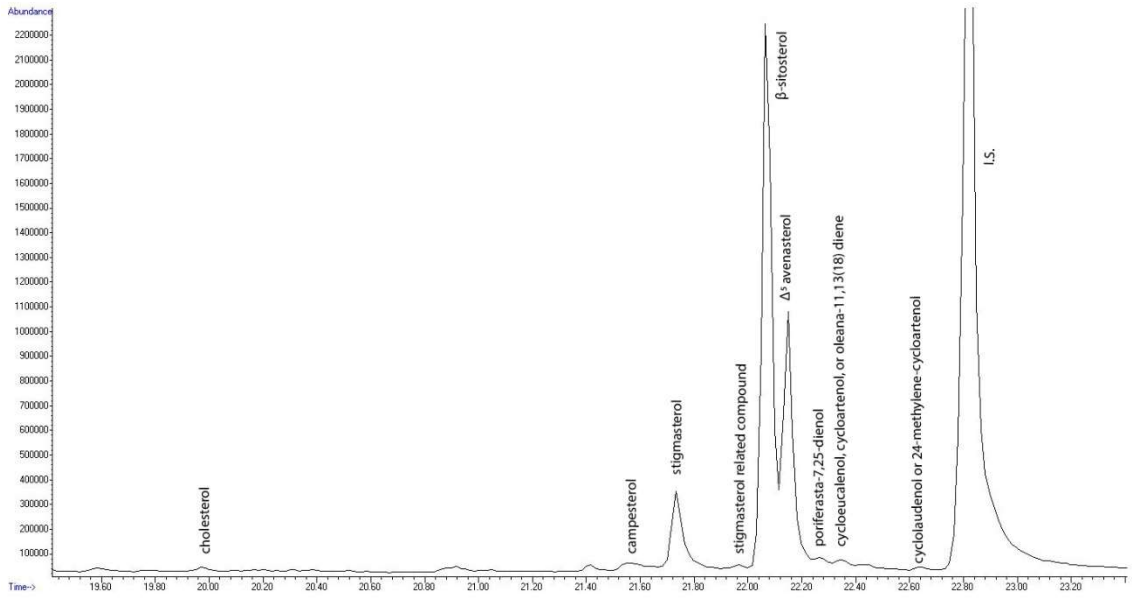

Figure.4.3 Partial sterol profile from the TLE of sample 5486

appears to be thicker on the base; it is certainly feasible that the slip prevented significant seepage into the base sample 7370. This could also be explained by a difference in sample size. The amount of sample taken was 1.2 g for 5486 and 0.6 g for 7370. Two times as much sample was taken at the body than at the base and over two times the amount of lipid was recovered in the body.

Summary

Different distribution patterns of lipids across the surface were observed according to the vessel type. The storage jar had consistently low overall lipids, while the bowl and open, handled vessel had significantly more. The low lipid amounts, lack of sooting, and the lack of ketones in the jar suggest liquid storage. Cooking in some form is suggested in the open vessel, while serving is suggested in the bowl. The sauceboat's lipid distribution was not very telling, but the likely absence of heat applied to the vessel does generally support the assumption that fine wares were not used for cooking.

Sample Pairs from Body

The rest of the vessels that were sampled in multiple locations were all from body sherds. The only group whose membership to the same pot is questionable is 7608, 11277, and 5448. These sherds were grouped by the project conservators as belonging to a reconstructed jar, but they did not directly mend to each other nor to the larger reconstructed pieces. Residue analysis could shed light on whether they belong to the same vessel. Many of these had above 100 µg/g in residue quantity, including the sample with the most residue in this study, 3133 A.

7489 and 3133 A

There is an enormous difference in quantity between pair 7489 and 3133 A, a difference of 985 µg/g! This difference is even more pronounced given that nearly three times as much sample was collected from the sherd with the lowest quantity, 7489, than 3133 A (3.2 g and 1.2 g, respectively). Sample 3133 A is exceptional in its quantity. It had pine resin and beeswax preserved. The evidence for the latter will be discussed in the Beeswax section. Any TAGs that were originally present are fully hydrolyzed, as evidenced by relatively small quantities of MAGs and DAGs and larger quantities of free fatty acids. Microbial degradation is evident with the odd-chain fatty acids from $C_{11:0}$-$C_{19:0}$ and branched alkanes and alkanols. Surprisingly for such an abundant residue, no sterols were recovered. Sample 3133 A seems to be a strongly insect-based residue mixed with plants. There does not seem to be as large of a plant presence, since all the major alkanes and alkanols, which are in substantially greater quantities than the rest, can be attributed to the hydrolysis of beeswax. Present also are α-ω diacids, specifically adipic acid (C6), azelaic (C9), and sebacic (C10). These diacids are fatty acid oxidation products which indicate that unsaturated FAs once existed in this sample (Copely et al. 2005; Mills and White 1994; Regert et al. 1998; Simic et al. 1992). Azelaic acid was narrowly the most abundant of the three diacids. The presence of $C_{9:0}$ suggests that $C_{18:1}$ was a compound in the original substance. These diacids and $C_{9:0}$ may indicate that the vessel held vegetable oil of some sort, possibly one that is not as high in phytosterols. Another substance was added, as indicated by range of fatty acids, but without sterols, it is difficult to determine the nature of the substance any further. Three interesting ketones, 2-decanone, 2 undecanone, and 3-dodecanone, were recovered from this residue.

Together this suite is found in rue (Mejri et al. 2010) and in *Commiphora rostrate* resin (McDowell et al. 1988). The latter is a shrub only found in the horn of Africa, so can be ruled out geographically (McDowell et al. 1988). The compounds that can be definitively attributed to plant and insect wax—alkanols, alkanes, wax esters, diols, and ketones—comprise 85% of the residue.

Sample 7489 is somewhat odd-dominated in its alkane distribution from C21-33, but it is not as pronounced as 3133 A. The alkane sequence peaked in two locations at alkane 23 and alkane 27. This looks like a plant alkane sequence that is partially degraded. The latter half of the alkane sequence mimics that in 3133 A. There was a small amount of cholesterol and a small range of FA, including $C_{18:1}$. There were no ketones, DAGs, or diacids as in 3133 A, but 15-hydroxy-7-oxo-dehydroabietic acid was found. Most likely, sample 7489 contained a mixture of degraded epicuticular wax from more than one plant, animal products, and pine resin. This sherd is rather thick and could have been part of a large base, which might explain its relatively low abundance of lipids. The vessel type to which this pair belongs is unknown.

3038 and 3292

Samples 3038 and 3292 are a pair of body sherds from a squat, wide-mouthed jar, roughly 13 cm in diameter. The amounts sampled were 1.6 g and 2.3 g, which yielded 207 µg/g and 165 µg/g of residue, respectively. Sample 3038 is likely degraded beeswax. Beeswax was clearly mixed with other substances, as MAGs, DAGS, and free fatty acids are present in the residue. Cholesterol was quite abundant in both the TLE and N, suggesting a significant animal component, with 3 µg/g in the neutral. Also present were the plant sterols β-sitosterol and germanicol in the neutral in small amounts. Germanicol

is found in many plants, including *Pistacia* resin (Assimopoulou and Papagerorgiou 2005). Although an unusual pentacylic triterpenoid, it cannot be assigned any further than to higher plants (Reber, Blitz, and Thompson 2010:48). There were numerous branched alkanols present in the neutral fraction, which is indicative of bacterial degradation. Also, 15-hydroxy-7-oxo-dehydroabietic acid was found. Both $C_{16:1}$ and $C_{18:1}$ and isomers of each were present. Fatty acid $C_{16:1}$ could indicate plants or fish, but cannot be resolved further. Exactly which $C_{16:1}$ and $C_{18:1}$ isomers were present could not be determined, because the extraction and derivatization protocol used does not allow for identification of the double bond position.

Sample 3292 initially appeared to contained beeswax also, but it is less certain. It contained all the wax esters attributed to beeswax, but in very small amounts. However, there seems to be evidence for their hydrolysis, as a large quantity of even alkanols 22-30 and of $C_{16:0}$ were present. Additionally, sample 3292 contained $C_{14:0}$ wax esters, indicating plant wax as well. Cholesterol, β-sitosterol, and germanicol were present with cholesterol as the most abundant sterol as in 3038. The pine resin degradation compound 15-hydroxy-7-oxo-dehydroabietic acid was also found. The FA ranged from $C_{9:0}$-$C_{28:0}$. The alkane distribution peaked at alkane 20, which was the highest alkane in the sample These sherds seem to belong to the same vessel, as they contain similar compounds, but this sample appears to be highly degraded.

8020 and 4469

Samples 8020 and 4469 belong to a tall, constricted neck jar with podes on the exterior. Both samples were tentatively identified as having beeswax. Beeswax was likely mixed with other substances, suggested by the wide range of fatty acids that are not

attributable to beeswax. Microbial degradation is evidenced by branched and odd-chain fatty acids and branched alkanols. Sample 4469 had $C_{18:1}$ and $C_{16:1}$. Alkanols dominated the TLEs of both, which likely indicates the hydrolysis of corresponding wax esters. DAGs and MAGs were present in both and indicate TAG breakdown, which again points to other substances mixed with beeswax. Pine resin markers were identified in both samples: DHA and 7,15-dihydroxy-dehydroabietic acid in trace quantities in 8020, and 15-hydroxy-7-oxo-dehydroabietic acid in 4469. In terms of sterols, 8020 had cholesterol, β-sitosterol, and Δ^5 avenasterol, and 4469 had cholesterol and β-sitosterol. All were found in small quantities of less than 1 μg/g and indicate animal and plant products. Both sherds were wiped in the interior prior to kiln firing. Also, interestingly, 8020 had a large amount (4 μg/g) of the compound tentatively identified as labdane.

4972 and 8096

Samples 4972 and 8096 are rather complex residues from a squat, wide-mouthed jar. They both yielded over 100 μg/g and contained very similar amounts of labdane. Sample 8096 had one of the largest amounts of wax esters in any sample (13.1 μg/g), yet none of them were the specific wax esters identified in beeswax. The fatty acid moieties were $C_{16:0}$, $C_{16:1}$, $C_{13:0}$, $C_{12:0}$, and $C_{14:0}$. There was a wide range of fatty acids in 8096 from $C_{8:0}$-$C_{26:0}$ even and $C_{9:0}$-$C_{17:0}$ odd. Unsaturated fatty acids ($C_{14:1}$, $C_{16:1}$, $C_{17:1}$, and $C_{18:1}$) were relatively abundant at 4.1 μg/g. $C_{16:1}$ was the most abundant within the unsaturated FA at 2.0 μg/g. The presence of $C_{9:0}$ indicates that there was more $C_{18:1}$ that had been oxidized. Cholesterol, its derivative, and oxidation product are present, indicating a strong animal component. Stigmasterol, β-sitosterol, and Δ^5 avenasterol indicate a plant component, which corresponds with the unusually high quantity of wax

esters. There were numerous saturated MAGs and DAGs, as well as branched alkanols and some branched alkanes. Microbial degradation is evident within these FA, alkanols, and alkanes. Unsaturated 20:1, 22:1, and 24:1 alkanols were present, in addition to the straight chain even-dominated alkanols. There was a wide range of alkanes, but there was a noticeable predominance of 21 and 23. The rest of the alkanes are all in low abundance. There was possibly one dicarboxylic acid—diacid 6.

Sample 4972 contained germanicol and DHA that 8096 did not have, as well as stigmastane. Both cholesterol and 7-ketocholesterol were identified. Sample 4972 contained a mixture of pine resin, plant epicuticular wax, and animal products with microbial degradation. The wide range of alkanols suggests more than one plant was present. The wide range of FA suggests multiple fatty sources from $C_{6:0}$-$C_{26:0}$, with the exception of $C_{7:0}$, $C_{21:0}$, and $C_{25:0}$. $C_{18:1}$ was present as a free fatty acid, bound in a MAG, and inferred from $C_{9:0}$. This sample also had $C_{16:1}$. It had wax esters with $C_{16:0}$ and $C_{16:1}$ moieties. Surprisingly, sample 4972 contained only a few wax esters. Similar to sample 8096 is the presence of adipic acid.

5448, 7608, and 11277

Samples 5448, 7608, and 11277 possibly belonged to the same coarse ware vessel, but this remains to be confirmed. Quantities varied from 6.2 µg/g, 25.9 µg/g, and 46.9 µg/g, respectively. The amount collected also varied: 2.8 g was collected from 7608, 2.5 g from 11277, and 1.9 g from 5448. Sample 5448 had a very small amount of residue, barely above the threshold for appreciable residues. It contained only OL 20 and 22 in the higher plant range and cholesterol. Sample 7608 had a mixture of plant and animal products with microbial degradation, especially of the fatty substances. These

include $C_{18:1}$ isomers and short-chain fatty acids from $C_{7:0}$-$C_{10:0}$. Two secondary alcohols, one wax ester, and a ketone possibly with 30 carbons were recovered in addition to small amounts of cholesterol, β-sitosterol, and stigmasterol. It only had alkanols 20, 22, and 24. The residues found in both samples 5448 and 7608 suggest one plant represented with the small range of alkanols.

Sample 11277 was quite different, especially in terms of a broader sterol content than 7608 and 5448. It contained cholesterol and campesterol in very small amounts. In larger quantities were stigmasterol, β-sitosterol, Δ^5-avenasterol, poriferasta-7,25-dienol, and possibly dihydrolanosterol. Furthermore, it had pine resin biomarkers, DHA and 15-hydroxy-7-oxo-DHA. Lastly, it contained the compound that was either oleana-11,13(18)-diene, cycloartenol, or cycloeucalenol. It had a broader range of alkanols than 5448 and 7608, which suggest the presence of more than one plant, and had a wider range of fatty acids than the other two sherds. It did not have obvious $C_{18:1}$ isomers or ketones.

It seems that 5448 and 7608 could plausibly have come from the same vessel, while 11277, with its mostly plant-based residue, does not seem to be from the same vessel. There are no obvious reasons for such different content, such as a slip preventing lipid absorption or the application of heat that could have destroyed lipids.

Handles

It has been suggested that handles could be used as a proxy for soil samples (Heron and Evershed 1993:256). This is a reasonable suggestion, given that one would not expect a handle to interact with the substance being cooked, processed, or served in the vessel enough to absorb appreciable lipids. It is, therefore, thought that any lipids

recovered from handles could theoretically indicate soil contamination. Testing handles for lipid residues can be methodologically useful when soil samples were not saved or accessible for residue analysis. Since the Ayia Triada excavation had been completed prior to this study being proposed and no soil samples had been collected for residue analysis, I decided to test these assumptions regarding handles.

I sampled four handles and analyzed two for this phase of the project, samples 8141 and 1656. Both had interpretable amounts of residue and a relatively large amount at that, 55.1 μg/g and 135.17 μg/g, respectively. Intriguingly, medium-coarse handle 1656 was a complex mixture of beeswax, pine resin, plant or fish fats, and animal resources. The range of fatty acids in this residue suggests a mixture of fatty substances. It had excellent preservation, as evidenced by short-chain fatty acids and glycerol in the TLE. Sample 8141, a coarse-ware handle, was a strongly plant-based residue with a minor animal component. It contained β-sitosterol, stigmasterol, and trace amounts of cholesterol. It was not particularly unsaturated with only $C_{18:1}$. The cholesterol and $C_{18:1}$ could be attributed to plants or animals, but both are present in such minor amounts that it is difficult to resolve. This sample had the look of degraded beeswax in terms of its alkane and alkanol sequence. However, it did not have any of the other main biomarker compounds nor their breakdown products.

Handles cannot be used proxies for soil. As these two samples show, they can be full of food residues. The quantity and content are considerably different than the lipids expected in soils (Heron, Evershed, and Goad 1991).

Fine Wares

Overview

Overall, I analyzed six fine-ware sherds. One sample had no lipids recovered, sample 8156. The mean quantity of residues from fine wares is 60.0 µg/g. Fine wares had the lowest residue mean of all fabric types, but also the fewest samples analyzed. Quantities ranged from 7.3 to 204.5 µg/g. Fine wares also encompassed the only non-local fabric types, a buff-ware pyxis sherd and two grey body sherds of unknown vessel type.

Fine-Ware Pyxis

One globular pyxis was sampled in this study, sample 129. Its fabric can be characterized as very fine, buff-colored, and was possibly painted. This fabric indicates it was an import (Mavridis and Tankosić 2016a:229). It yielded 76 µg/g of residue. Cholesterol and its oxidation product and β-sitosterol were recovered in quantities above trace levels, suggesting degraded animal fats and plants. The alkanols and alkanes were highly branched, suggesting significant microbial breakdown. There was a limited range of FA, from $C_{14:0}$-$C_{18:0}$ even, $C_{15:0}$, $C_{16:1}$, and $C_{18:1}$. The quantity of MAGs was two times the amount of FFA, suggesting that the acylglycerols are not as completely hydrolyzed as some other samples. The alkane sequence likely represents epicuticular wax from one plant. There are many secondary alcohols in the neutral fraction, between OL 14 and OL 22. These originate from plant or insect waxes (Kolattukudy 1976). It is apparent that this sample contained degraded plant or insect wax mixed with animal fats. No pine resin was found.

Fine-Ware Sauceboats

One definite fine-ware sauceboat was analyzed, sample 2062, while another possible one was analyzed with two samples, 5486 and 7370. The latter sauceboat has already been discussed in the Lipid Distribution Studies section. Sample 2062 contained a large quantity of sterols much like sample 5486. It contained a total of 204.5 μg/g residue, 6.5 μg/g of which were sterols. Cholesterol, its oxidation product, 7-ketocholesterol, and its derivative, cholesta-3,5-dien-7-one, were extracted from the TLE and N fraction. In all, cholesterol or cholesterol-derived compounds total 3 μg/g of the TLE and are roughly equal in quantity to the three plant sterols, stigmasterol, β-sitosterol and Δ^5 avenasterol. The presence of animal products is rather surprising, given one proposed function of this vessel related to drinking practices (Fahy 1964). Also, pine resin was identified with 15-hydroxy-7-oxo-dehydroabietic acid. This sample had a wide range of alkanols from 22-32, which suggests more than one plant was present. The alkanes display a distribution centered on alkane 24, the same pattern found other samples in this project. This pattern in addition to highly branched alkanes and alkanols, branched fatty acids, and odd-chain fatty acids suggests significant microbial degradation. Saturated DAGs were scarce, but MAGs were relatively more abundant. Interestingly, almost a quarter of the free FA are unsaturated. Specifically, $C_{18:2}$, $C_{18:1}$, and two isomers of $C_{16:1}$ were found in the TLE. FA $C_{8:0}$-$C_{10:0}$ could indicate $C_{18:1}$ breakdown, although the quantities of these short-chain FA are very small (Mills and White 1994:40). This could signal that this vessel contained degraded vegetable oil, which will be discussed in detail in Chapter Five. Furthermore, two diacids were found--

adipic acid (C6) and possibly pimeic acid (C7). Many secondary alcohols were found in the neutral section, roughly 4% of the neutral or 3.5 μg/g. This suggests plant or insect waxes with the former being most likely because of the phytosterol content. The residue in sample 2062 can be interpreted as a mixture of plant and animal resources with pine resin.

Fine-Ware Unknown

Three samples were taken from fine-ware body sherds of unknown vessel types—samples 7219, 7078, and 8156. These were the only sherds besides 129 that were not made with local clays, which signals that they were imports. The ceramic matrix of all three is a fine, grey fabric. Sample 8156 was the only slipped sherd, which may explain its negligible residue quantity. A total lipid content of 24 μg/g in 7219 and 29 μg/g in 7078 was recovered from these sherds. Sample 7219 was apparently comprised entirely of plants. It had a trace amount β-sitosterol, but no other sterols. It contained a small range of fatty acids from $C_{12:0}$-$C_{18:0}$ even in the TLE and some saturated MAGs, but in very small quantities. Alkanes dominated the sample and spanned the full range from C20-33. They peaked at C24 and C26 and contained many branched alkanes between C24 and C29. The alkanols, on the other hand, had a tighter range with only C20, C22, C24, and C26. The range of alkanols was smaller than in most samples and does not seem to be degraded.

Sample 7078 had a very similar alkane and alkanol range as 7219. It had a wide alkane sequence from C20-33 and peaked generally with the same alkanes. Branched alkanes were identified around the C26-29 range only, indicating slight degradation. The

alkanols ranged from C22-28 even and were not abundant overall. This sample too had a small quantity and range of free fatty acids, $C_{14:0}$, $C_{16:0}$, and $C_{18:0}$, and a small amount of DAGs and MAGs. Trace amounts of cholesterol and β-sitosterol comprised the sterol profile. Whereas 7219 contained only plants and perhaps a small range of them, 7078 was similar only with the addition of some cholesterol (0.28 μg/g). Interestingly, there were no wax esters in either of these. One would expect at least some in such a plant-based residue as 7219 or at least abundant hydrolysis products, which were also not present. Although 7219 and 7078 share the same unique fabric type, they may or may not belong to the same vessel and/or type.

Medium-Coarse Wares

Overview

I sampled 30 medium-coarse sherds, 13 of which were from known vessel types. The mean residue quantity was 101 μg/g. This group encompassed the largest number of samples. Only one sherd displayed an imported fabric type, sample 7063.

Medium-Coarse Jars

I collected seven samples from medium-coarse jars, representing five jars in total. Sample 7538 was a pithos neck that was found to be interpretively empty. As discussed in detail above in Lipid Distribution Studies, 3038 and 3292 belonged to the same jar while 4469 and 8020 belonged to another jar. Both pairs had pine resin, plant and animal products, and tentatively identified beeswax.

Samples 5025 and 7063 did not have evidence for beeswax or pine resin like the two jars above. Sample 5025 had a relatively large amount of unsaturated FA, including $C_{16:1}$, $C_{18:1}$, and $C_{18:2}$. Fatty acids $C_{18:2}$ and $C_{16:1}$ could indicate fish or plants. It exhibited a wider range of sterols than any of the medium-coarse jars that included cholesterol, 7-ketocholesterol, and three common plant sterols. Sample 7063 contained only cholesterol, but it had evidence for plant wax from more than one plant. It also had a series of unidentified compounds with peaks at m/z 73, 117, 131, and 175 that eluted between alkanol 17 and alkanol 21. It had a large amount of secondary alcohols as well. Neither sherd had particularly large amounts of residue, with 25 µg/g for 7063 and 50 µg/g for 5025. Both sherds were comprised of a mixture of plant and animal products.

Medium-Coarse Bowls

I collected samples from six sherds belonging to five medium-coarse bowls. Samples 3040 and 15787 were from the same bowl and were discussed already in the Lipid Distribution Studies section. The other sherds were 8428, 8092, 7599, and 5175. Sample 5175 was more complex than the others and had a significantly larger quantity at 458.7 µg/g. It is discussed more in depth in the High Yield Residue section. Samples 8428, 8092, and 7599 were all generally animal- and plant-derived residues with cholesterol and plant sterols β-sitosterol or Δ^5 avenasterol. None of these bowls had pine resin or beeswax. They all had appreciable amounts of secondary alcohols.

Medium-Coarse Unknown

A total of 17 samples were collected from sherds of unknown vessel type: six bases, four body sherds, two rims, one shoulder, one handle, and three base/body sherds. Three had evidence for beeswax: 1656, 5475, and 6683. The former was a handle and the other two were bases. Two samples were indeterminate with regard to their lipid content: 5489 and 7064 A.

Over a third of the residues in this category were heavily plant-derived, based on sterol biomarkers, alkane and alkanol distributions. Samples 2991, 5112 A, and 12461 had almost the exact same sterol profile: a series of unusual plant sterols alongside the more ubiquitous phytosterols. They either had trace cholesterol or none at all. These will be discussed in detail in the High-Yield Residues section. Samples 8072 and 7064 B were made up of mostly plant resources as well, but contained the more ubiquitous plant sterol profile. Samples 2991 and 5112A had higher unsaturated FA levels with $C_{18:2}$, $C_{18:1}$, and $C_{16:1}$. Unsaturation fatty acid levels of the rest were more similar, with the presence $C_{18:1}$ and/or $C_{9:0}$.

One sample of this group contained mainly animal products, 3275. Cholesterol was present in a minute amount, although this sample generally did not have a substantial quantity of residue to begin with at 18.2 μg/g. It contained a limited FA and MAG range, none of which were unsaturated. Its alkane range was even-dominated and showed evidence for degradation.

Five sherds from the medium-coarse unknown wares revealed mixtures of animal and plant resources: 3198, 5278, 5905, 7184, and 10421. All contained biomarkers for animals, albeit in trace amounts in some, while all except 3198 contained biomarkers for

plants with β-sitosterol and/or Δ^5 avenasterol. Even though sample 3198 did not contain plant sterols, the presence of plant wax is inferred from its odd-dominated alkane and even-dominated alkanol distributions. The alkane and/or alkanol distributions of all except 5905 suggest more than one plant. Samples 5905 and 10421 also contained one unsaturated FA, $C_{18:1}$. There is no way to tell if these ingredients were used separately or as a mixture (Evershed 2008b:27; Heron and Evershed 1993:258). All five of these samples were well below the mean for medium-coarse wares, ranging between 13.4 µg/g and 38.7 µg/g.

One sample, 14750, contained a complex mixture of plant wax, meat, and pine resin. Animal products are indicated by cholesterol and 7-ketocholesterol. The presence of 15-hydroxy-7-oxo-dehydroaboetic acid indicates degraded pine resin. There is a small quantity of Δ^5-avenasterol. Saturated MAGs/DAGs were not abundant. Fatty acids extracted in the TLE were: $C_{9:0}$, $C_{12:0}$-$C_{18:0}$, $C_{16:1}$, and $C_{24:0}$. $C_{15:0}$ and $C_{17:0}$ had multiple branched isomers, in addition to their straight chain counterparts. Alkanes were relatively abundant, making up 49% of the TLE. The full sequence from 18-33 was present and many were branched. The alkanols were even-dominated from alkanols 20-30, some of which were branched. Minor amounts of two $C_{14:0}$ wax esters and one $C_{16:0}$ wax ester were recovered.

The three sherds with beeswax (1656, 5475, and 6683) had residue quantities well above the mean for medium-coarse wares from 133.6 µg/g to 596.7 µg/g. Sample 6683 contained the highest quantity of the three. Although all three contained beeswax, animal, and plant-derived compounds, differences were observed. Sample 1656 had pine resin while the others did not. While all contained cholesterol, sample 1656 also had its

oxidation product. Sample 6683 had $C_{18:2}$ and $C_{16:1}$, which could suggest fish or plants. Samples 5475 and 6683 had $C_{18:1}$ isomers. Sample 6683 was a more complex mixture than the other two, as it also contained ketones with 29 and 31 carbons and diacids C6, 8, 10, and 11.

Coarse Wares

Overview

A total of 28 coarse-ware samples were collected, 13 of which were from known vessel shapes. The mean quantity of residue for coarse wares was 149.5 μg/g. Some of the most complex and most concentrated residues were coarse wares, in particular 3133 A, 6682, 7056, and 7623. All of the coarse wares were made with local fabric types.

Coarse Jars

A total of 10 samples were taken from coarse-ware jars. Two pairs (4972/8096 and 5436/7684) were collected from the same respective jars. These were all body sherds, except sample 7684. This sample was a rim and was the only coarse-ware jar sherd found to be interpretively empty.

Samples 7608, 8506, and 5448 were generally similar, although they differed in residue quantity. Animal and plant derived residues were recovered from all three. Sample 5448 contained very little residue; however, its cholesterol suggests animal products and alkanols 20 and 22 suggest wax from one plant. Sample 7608 was comprised of plant sterols, including β-sitosterol and stigmasterol, wax esters, and a wider range of alkanols, which suggests more than one plant. It also contained two $C_{18:1}$

isomers and straight and branched chain $C_{15:0}$ and $C_{17:0}$. Sample 8506 contained cholesterol like 7608 and 5448, but also contained its oxidation product. It did not have any plant sterols. Plant wax is inferred from the wax esters, α-ω diols, alkanols C22-26 even, and a series of alkenes (C19:1, C20:1, C21:1, C23:1). It also had short chain fatty acids $C_{6:0}$ and $C_{9:0}$.

Two jar sherds (1686 and 5436) had slightly more unsaturated fatty acids, specifically $C_{18:2}$, which could indicate a stronger plant or fish presence. They also contained a mixture of animal and plant wax. The range of alkanols suggests multiple plants. Sample 1686 had a small range of alkanes, unlike 5436. However, its neutral portion was unusable due to the contamination issues discussed earlier. It is likely that more alkanes existed in the neutral portion of this sample that had to be discarded. Of interest, sample 1686 was the imported yellow-mottled ware.

Two other jar sherds showed evidence for pine resin, samples 11277 and 4972, but otherwise their residues are dissimilar. Sample 11277 had a wide range of sterols previously discussed the Lipid Distribution Studies section and could be a solely plant-based residue. Sample 4972 contained a mixture of plant resin, plant wax, and animal lipids. It came from the same jar as 8096; however, 8096 did not have pine biomarkers. The pair 4972 and 8096 were also discussed more in depth in the Lipid Distribution Studies section.

Sample 7056 was the only coarse-ware jar that contained both beeswax and pine resin. Beeswax was clearly mixed with other substances given the fatty acids present. Residue originating from plants is evidenced by β-sitosterol. It had unsaturated alkanols C20:1 and C22:1. Straight and branched chain $C_{15:0}$ and $C_{17:0}$ indicate microbial

breakdown of the fatty substances. Two isomers of both $C_{16:1}$ and $C_{18:1}$ were also present. Sample 7056 also contained diacids 14 and 16, 2-decanone, valeric acid or succinic acid, and 4-hydroxybutryic acid.

Coarse Bowls

Only one coarse-ware bowl was sampled, 5510. The sample was taken from its incurving rim. The quantity of lipids was rather small at 9.3 µg/g. It only had evidence for plants, including degraded epicuticular wax, as indicated by β-sitosterol and the alkanol distribution. No unsaturated FA were detected.

Coarse Other Shapes

An open, handled vessel was sampled in two areas: at the base/body with sample 11309 and on the body with 3482. As discussed in the Lipid Distribution Studies section, this vessel is thought to be a cooking vessel due to its heat biomarkers and distribution of lipids.

Coarse Unknown

A total of 15 samples were taken from coarse-ware sherds of unknown vessel type. Nine were body sherds, four were bases, one was a rim/body, and the last was a handle.

Only one of the coarse sherds seemed heavily plant-based, sample 5217. This body sherd contained only one FA ($C_{16:0}$) and plant sterols β-sitosterol, stigmasterol, and Δ^5 avenasterol. The alkane and alkanol sequence suggest a limited number of plants; however, the neutral fraction of 5217 was unusable. It is possible that a wider range of

sterols, alkanes, and alkanols existed in residue and would have been extracted into the neutral fraction.

Eight of the coarse-ware sherds from unknown vessel types had pine resin in them: 3133 A, 7623, 7760, 5114, 5665, 7489, 3049, and 8849. Only one of these with pine resin also had beeswax, sample 3133 A, and it was the highest yielding residue of the entire sample set. Sample 7623 is also a quantity outlier and will be discussed in detail in the High Yield Residue section. Four of the remaining samples were actually quite similar: 7760, 5114, 5665, and 7489. They all had degraded plant and animal mixtures with signs of microbial degradation. Their alkane and/or alkanol sequences suggest more than one plant resource was present. Samples 7760 and 5665 had $C_{18:1}$ isomers and straight and branched chain $C_{15:0}$ and $C_{17:0}$. Sample 5665 and 5114 contained $C_{16:1}$, indicating plant or fish. Sample 5114 contained wax esters, and secondary alcohols.

Samples 3049 and 8849 contained pine resin, but other compounds set them apart from the ones above. They contained meat and multiple plant sources. In addition, sample 3049 yielded stigmasta-3,5-dien-7-one. It was well preserved, as indicated by the short chain fatty acids. Sample 8849 had the diacid succinic acid and laevulic acid, as well as $C_{18:2}$. The latter could be attributed to plants or fish. This was a rather diverse residue, which is surprising given how little reside was recovered (15.5 µg/g). It too was well preserved, as indicated by $C_{6:0}$-$C_{9:0}$ and succinic acid. This sample is imported yellow-mottled ware.

Three unknown vessel sherds did not have any evidence for pine resin or beeswax, samples 3035, 5920, and 8141. Sample 3035 was well preserved and contained evidence of heated fatty materials with ketones 31 and 33. Sooting on the interior

corroborates the ketone evidence. It also had another ketone, 2-heptadecanone, and a similar length diol, n-heptadecan-1,2-diol. A mixture of plant epicuticular wax, and animal resources characterized this residue. Two $C_{18:1}$ isomers were present. Various wax esters were present with $C_{10:0}$, $C_{14:0}$, and $C_{16:0}$ fatty acid components. Fatty materials probably originated from multiple sources. Multiple plant sources were represented. Good preservation was indicated by the presence of $C_{8:0}$-$C_{10:0}$ and adipic acid. Sample 5920 had evidence for only animal products and limited plant resources. Alkanols 22-26 even were the only recovered from this compound class that can be assigned to the higher plant range. Sample 8141, the handle, contained a small amount of cholesterol in addition to more abundant plant sterols of β-sitosterol and stigmasterol. The saturated fatty acids $C_{16:0}$-$C_{24:0}$ even were present.

Sample 6682 yielded the second highest lipid quantity in the present study at 915 µg/g. Beeswax dominated the residue, but it was clearly mixed with plants and other degraded fatty substances. It had only one MAG, but a range of fatty acids from $C_{12:0}$-$C_{24:0}$ (except $C_{23:0}$). It had $C_{14:1}$, $C_{15:1}$, $C_{16:1}$ (in two isomeric forms) and $C_{18:1}$ (in two isomeric forms). It also had unsaturated OL 28:1 and numerous ketones. In fact, this sample yielded the most ketones of any sample at 32.3 µg/g. It had 16-hentriacontanone, 16-pentacosanone, 16-hexacosanone, 16-heptacosanone, 16-octacosanone, 16-nonacosanone, nonacosan-25-one, and 15-nonacosanone. It also contained diacids C4-8, 11, and 14. The odd-chain diacids were less abundant than the even counterparts in the neutral fraction. The diacids were previously bound, since they only appeared in the neutral fraction. Also, it had the following compounds not found in any other sample from this study: glycolic acid, 2-furoic acid, β-lactic acid, and 5-oxy-valeric acid.

Surprisingly absent compounds, given the copious amount of residue recovered, were sterols. Sterols were also absent from a similarly abundant residue, sample 3133 A.

The last two samples yielded no information. Sample 7183, a coarse-ware base, was found to have negligible residues. Sample 653 had appreciable residues, but it was impossible to interpret; it had no sterols or other biomarkers and its neutral component was unusable.

Beeswax

Pure beeswax has been subjected to GC/MS by numerous authors (Charters et al. 1995; Heron et al 1994; Regert et al. 2001; Tulloch 1971; Tulloch and Hoffman 1972 and many others). Beeswax is identified by a series of homologous compounds: odd alkanes from C23-C35 with a peak at C27, even alkanols from C24-34, small quantities of even fatty acids from $C_{22:0}$-$C_{36:0}$ with $C_{24:0}$ being the most abundant, and palmitic acid wax esters with C40-50 peaking at C46 (Tulloch 1971; Tulloch and Hoffman 1972:698-699). Present in smaller amounts are wax esters with $C_{18:0}$, $C_{18:1}$, and $C_{20:0}$ fatty acid moieties (Tulloch 1971:245). Less often cited are a series of α-(ω-1) diols with 24-32 carbons and minor amounts of associated α-ω diols; the major α-(ω-1) diols from this range are 1,23 tetracosanediol, 1,25 hexacosanediol, and 1,27 octacosanediol (Tulloch 1971:238). Heron and colleagues (1994:267) recovered five additional homologous coeluting compounds between the wax esters with mass spectral peaks of m/z 117 and 131 in both modern and archaeological beeswax. The compound with the peak m/z 117 could be a secondary alcohol. Other minor compounds in beeswax are odd-chain alkenes, which are merely unsaturated alkanes, of which 29:1, 31:1 and 33:1 are the most abundant (Maia and

Nunes 2013:963). Two minor unsaturated alkanols have also been found in beeswax, dotriaconten-1-ol (C32:1) and tetratriaconten-1-ol (C34:1) (Jiménez et al. 2003:112).

Archaeological time alters the compound distributions observed in fresh beeswax, as well as the application of heat during use. Heron and colleagues (1994:268) demonstrated experimentally that heat can lead to the complete disappearance of alkanes, although they reported that the wax esters remained stable. Regert and colleagues' (2001:561) accelerated aging experiments showed a decrease in the abundance of smaller carbon alkanes, which shifted the overall alkane distribution. Archeologically, this pattern was confirmed in samples from Dikili Tash and an Egyptian Fayum portrait, in which alkane 27 was much depleted and therefore, alkane 31 predominated (Regert et al. 2001:563). Furthermore, the hydrolysis of wax esters during the use of the vessel, by heat, or as a result of burial yields palmitic acid ($C_{16:0}$) and long-chain even alkanols from C26-C34 with 1-triacontanol dominating (Charters et al. 1995:119-120; Evershed at al. 1997:981; Regert et al. 2001:560).

Six samples in this study have compelling evidence for beeswax: 1656, 3133 A, 5475, 6682, 6683, and 7056. The gas chromatogram for the TLE of sample 7056 is shown (Figure 4.4). These samples displayed an odd-dominated alkane sequence, but there were subtle differences in their relative abundances (Figure 4.5). The samples in which n-heptacosane (C27) is the most abundant and therefore, those that more closely resemble the alkane distribution of fresh beeswax are 7056, 3133 A, 6682, and 6683. Sample 5475 and 1656 show a depletion of alkane 27. The alkane distribution of these samples mirror that of Regert and colleagues' (2001) experiment. The wax ester sequence, much like the alkane sequence of samples 5475 and 1656, differs in relative

abundance from fresh beeswax. Although all contain wax esters with C40-48, none display the same pattern in modern beeswax with C46 as the most abundant of the esters. Samples 3133 A, 6682, and 6683 peak at wax ester 40 and progressively decrease in abundance to wax ester 48. Samples 1656 and 5475 have the proper range of wax esters, but all in very low and roughly equivalent quantities. In sample 7056, wax ester 40 is the most abundant, and 42 and 46 are the next most abundant. The appearance of massive amounts of alkanols in all these samples show that the wax esters were almost completely hydrolyzed. Alkanol 1-triacontanol is the most abundant within this compound class in samples 1656, 3133 A, 5475, and 7056. This provides strong evidence for the original predominance of C46. Sample 6683 had OL 28 with the largest amount, followed by OL 30. Sample 6682 is the only sample where OL 30 is not one of the most abundant alkanols; instead, OL 24, 26, and 28 were the three most abundant alkanols in this sample.

Besides the alkane and wax ester sequences, these samples have other compounds that provide supplemental support for beeswax. The samples had some (or all, in the case of 7056) of the five compounds with a base peak of m/z 117 between the palmitic wax esters. They all contained wax esters with $C_{20:0}$ fatty acid moieties, substantial $C_{16:0}$, and large amount of $C_{24:0}$. Samples 1656, 3133 A, 5475, 7056, and 6682 contained the entire sequence of α-(ω-1) diols found in beeswax. Sample 6683 contained three of the diols: 1,27 octacosanediol, 1,29 triacontanediol, and 1,31 dotriacontanol. Furthermore, all except 3133 A had unsaturated alkanols C32 and/or 34. Sample 3133 A had alkene 33:1.

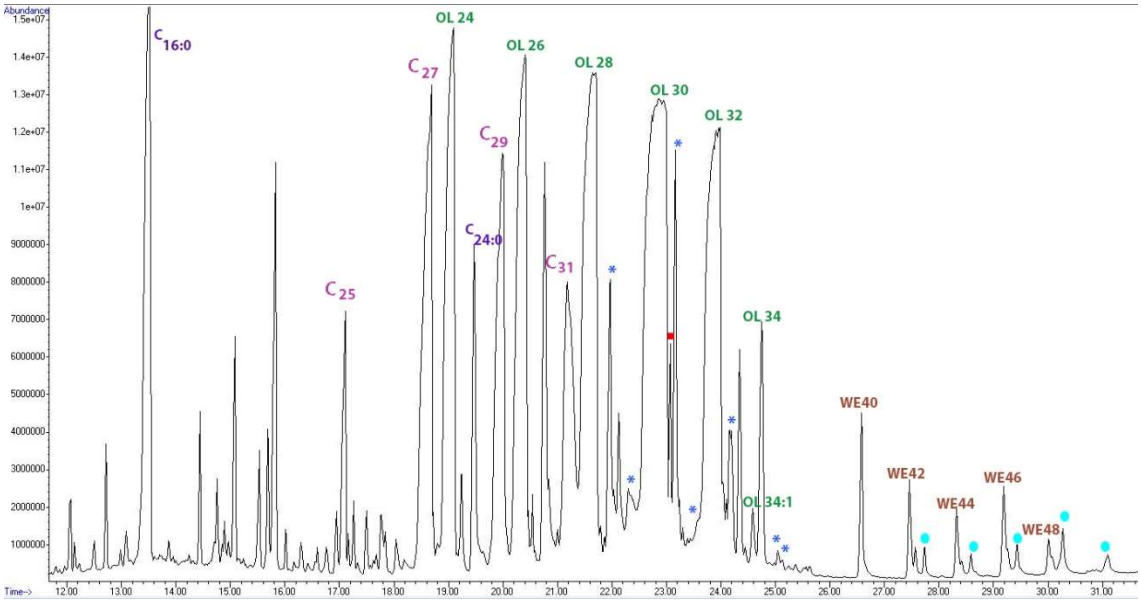

Figure 4.4 Gas chromatogram of sample 7056 with beeswax. [X=carbon number; $C_{x:0}$ = saturated fatty acid; Cx= alkane; OLx=alkanol; OL 34:1= unsaturated alkanol; asterisk=α-(ω-1) diols; WEx= wax ester; blue circle=compound with m/z 117 peak; red square= internal standard (n-TTC)]

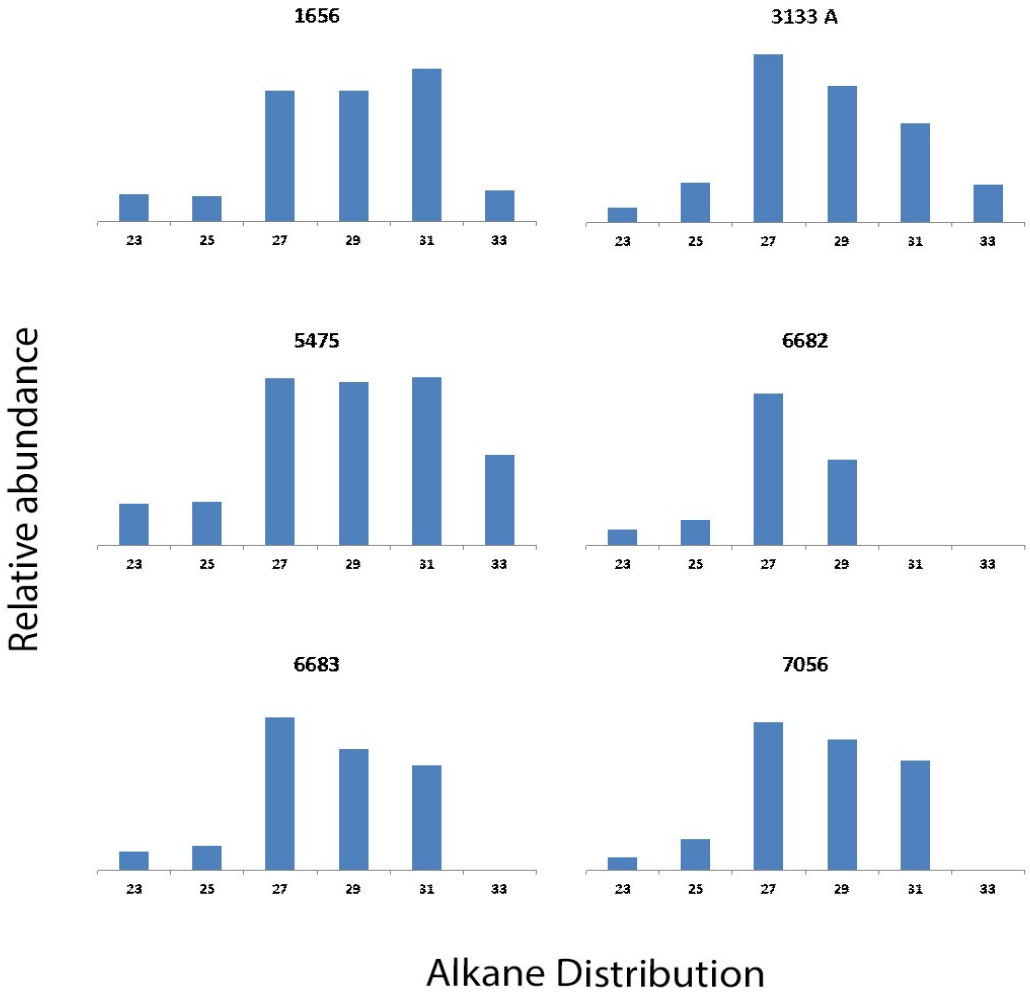

Figure 4.5 Alkane distribution of samples with beeswax

In these six samples, therefore, beeswax is confirmed by numerous compound classes.

Three other samples likely contained beeswax—3038, 8020, 4469—but it was highly degraded. There appeared to be loss of some minor compounds that provide supporting evidence for beeswax in the samples above. The latter two sherds belong to the same vessel. The alkane sequences with alkane 31 predominating is the same observed in Regert and colleagues' (2001) study (Figure 4.6). The larger amount than expected of alkane 23 in samples 3038 and 4469 could indicate the addition of a plant wax. All had the proper alkanol, wax ester, and fatty acid constituents, but were not in the exact distribution of fresh beeswax. Samples 8020 and 4469 look identical with an alkane sequence that peaks at 31, abundant alkanols with the highest alkanols of 28, 30, 32, and the wax ester predominance of WE 44. They also have low amounts of $C_{22:0}$-$C_{30:0}$ with $C_{24:0}$ as one of the most abundant. Since the wax ester sequence differs from that of fresh beeswax, other compounds would be needed to make a more secure identification. Unfortunately, the diols nor the compounds with m/z 117 peaks were found in these samples. Sample 3038 had the same alkane sequence with a peak at 31, wax esters that decrease from 40 to 48, 1,23 tetracosanediol, and long chain alkanols. The alkanol sequence does not peak at OL 30, however. This sample contained a few of the proper fatty acids, $C_{20:0}$, $C_{22:0}$, and $C_{24:0}$. These three samples are not quite as secure of identifications as the six above, but are probable.

Sample 3292 belongs to the same vessel as 3038 and initially, it looked like it also contained beeswax. However, too many of its compounds do not quite match the proper distribution and/or have the proper constituents. Its alkane sequence is odd-dominated, but does not reflect the preferential depletion of lower carbon alkanes. It had all of the

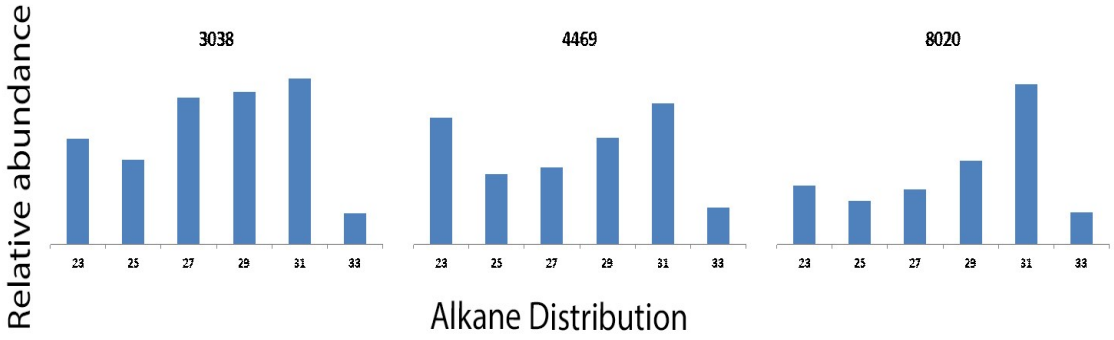

Figure 4.6 Alkane distribution of samples with possibly degraded beeswax

palmitic wax esters as beeswax, albeit in low amounts, and the proper fatty acid range with $C_{24:0}$ the most abundant. The low wax esters could be explained by more complete hydrolysis, in which case one would expect high levels of $C_{16:0}$ and even carbon number alkanols. However, this was not the case. Although the $C_{16:0}$ level was high, there was only a small amount of long chain alkanols. Alkanols 24, 26, and 28 were in greater abundance than the expected 30. The quantities of the alkanols are not substantial enough to warrant this explanation. Without corroborating evidence, it cannot be confirmed as beeswax.

In terms of vessels, most of these are from unknown types. The four samples from known vessel types belong to three medium-coarse or coarse jars. When the nine samples with beeswax are grouped by sherd type, we see that most are body sherds. Three are base sherds, five are body sherds, and one is a handle. The best examples are from levels 4, 5, and 5a. Half of the best-preserved examples are from bases. These pots all contained a mixture of beeswax and other substances.

High-Yield Residues

Several samples stand out as having either an exceptionally high residue quantity above 350 µg/g and/or a unique sterol profile. The latter especially warrants further discussion, because sterols tend to be diagnostic compounds and one of the least common compound classes. The quantity outliers, in decreasing order, are 3133 A, 6683, 6682, 5175, 7056, and 7623. Samples 3133 A, 6682, and 7056 have already been discussed in detail in several other sections.

Sample 7623 was a flared rim/body sherd from a coarse unknown vessel. It contained 373.6 µg/g of lipid. A significant amount of the TLE (70%) consisted entirely of FA, which signifies mass hydrolysis of acylglycerides. However, the FAs were not terribly diverse. They ranged from $C_{12:0}$-$C_{20:0}$ (except $C_{19:0}$) and were heavily saturated. A significant amount of $C_{16:0}$ and $C_{18:0}$ was observed, including underivatized $C_{18:0}$. Only 3% of the total fatty acid content was unsaturated, all of which belonged to $C_{18:1}$. Branched $C_{15:0}$ and $C_{17:0}$ were present. The MAGs and DAGs were relatively abundant and exclusively saturated. Pine resin biomarkers,15-hydroxy-7-oxo DHA and DHA methyl ester, were found in this sample. DHA methyl ester was found in the blank in 0.8% relative to the amount in the sample; such a low relative amount can justifiably be subtracted out. Sterols included cholesterol, 7-ketocholesterol, cholesta-3,5-dien-7-one, β-sitosterol, and stigmastanol. A small quantity of wax esters with $C_{16:0}$ and $C_{18:0}$ FA components were extracted. A substantial quantity of $C_{16:0}$, 122.6 µg/g, could indicate the hydrolysis of many more wax esters. It comprises a third of the TLE. Alkanes were odd-dominated with 29 and 31 predominating; some alkanes were branched. There was a

series of short-chain alkenes present, and alkanols were even-dominated from 22-30. Many branched alkanols were also present. Abundant mostly straight chain diols were present: 1,2-heptadecane 1,2 diol, 1,2-octadecane-1,2 diol, 18-methyl-nonadecane-1,2-diol, 20-methyl 1-2 diol docosane, and 22-methyl 1,2 diol tricosane. Ketones included heptadecane-2-one and K31. Taken together, these compounds suggest a complex mixture of pine resin, animal, and plant products, including epicuticular wax, with microbial degradation. Multiple plants were represented from the range of alkanols and alkanes. The oxidized cholesterol and the overall branched nature of the residue might be explained by the position of this sample at the rim/body, which would have been more exposed to oxygen (Reber et al. 2015). Ketone 31 could indicate the application of heat, but this interpretation would need to be shored up by the presence of ketone 33 and 35 also (Evershed et al. 1995; Raven et al. 1997).

Over 458.7 μg/g was extracted from another high yield sample 5175. This sample contained a high sterol content at 7.1 μg/g. Sample 5175 is a medium to large bowl. Unfortunately, this was one of the residues for which the associated neutral was not useable. This was a complex, waxy mixture of plant, animal, and possibly resin. Sample 5175 had the highest amount of the labdane-type compound. The range of fatty acids is quite small ($C_{8:0}$-$C_{10:0}$, $C_{12:0}$, $C_{14:0}$, $C_{16:0}$, $C_{17:0}$ branched) and is dominated by $C_{14:0}$. Second and third in abundance are $C_{12:0}$ and $C_{17:0}$ branched. Surprisingly, there was no $C_{18:0}$. Although there were no free unsaturated fatty acids, $C_{18:1}$ is indicated by the presence of $C_{9:0}$ and is bound in a MAG along with $C_{16:1}$. The fatty acids do not show intense microbial degradation. No DAGs were preserved.

Other compounds include secondary alcohols, alkanes, alkanols, and sterols. Numerous secondary alcohols were recovered; a secondary alcohol is the second most abundant compound in the TLE. Alkanes and alkanols total 58% of the residue. Alkanes ranged from C18-33, peaked at C24, and were highly branched. There was a wide range of alkanols from OL 22-34, with OL 30 being the most abundant. Alkanols too show signs of microbial degradation. Interestingly, the alkanols displayed the distribution for hydrolysis of beeswax wax esters and some C40-48 wax esters were recovered with the $C_{16:0}$ fatty acid component. However, the alkane sequence was not odd-dominated as it should for beeswax. Unsaturated alkanols 20:1, 22:1, and 24:1 were present. Sterols include cholesterol and its derivative cholesta-3,5-dien-7-one, stigmasterol, β-sitosterol, and Δ^5 avenasterol. The sterol β-sitosterol was found in the TLE blank, but in a small amount relative to the total quantity amount in the sample. Phytosterols totaled slightly more at 3.9 µg/g than cholesterol or its derivatives, which totaled 3.2 µg/g. The compound o-coumaric acid was found only in this sample. It could originate from several sources, including olives, peas, oregano, and fennel (Duke 1992). The residue from this bowl seems much more complex than the rest of the bowls. Although the sterol content is high, the sterols are no more diagnostic than general plant and animal.

Sample 6683, a medium-coarse base, contained a significant amount of residue at 596.7 µg/g. Beeswax is mixed with plants, as β-sitosterol is abundant. Only $C_{15:0}$ branched suggests microbial breakdown of the FA. The unsaturated FA $C_{18:2}$ indicate either fish or plants. Sample 6683 contained two $C_{16:1}$ and three $C_{18:1}$ isomers. Surprisingly, given the abundant overall residue, the quantity of MAGs/DAGs was low. It had two ketones (16-nonacosanone and K31), diol 1,2 nonacoane, and diacids 6, 8, 10,

and 11. It also possibly had hydroxyl FA or oxo FA in the TLE. It was well preserved and showed minimal microbial degradation. Numerous unidentified compounds were found in this sample.

Several samples contained an unusually high sterol quantity and in most, an unusual sterol content as well. Samples 12461, 5175, 2062, 2991, and 5486 yielded between 6.0 and 12.0 μg/g of sterols. Three of these have already been discussed earlier in this section or in other sections, 5175, 2062, and 5486. The sterol content of 2062 and 5175 is high in quantity, but not unusual in content like 5486. The remaining high sterol sherds, 2991 and 12461 will be discussed here.

The most startling example is 12461. It only had 38 μg/g total residue, yet a third of the quantity belonged to sterols, typically the least common compound class. Alkanes present were 20 and 23-31, with a clear dominance of alkane 29 and 31. Alkanols were present in a tight range with C20, C22, and C24 in very small abundance. The fatty acid range is limited from $C_{14:0}$-$C_{20:0}$ even with the addition of $C_{18:1}$, as well as scarce saturated MAGs and one possible DAG. There were no branched fatty acids and only very minor branching within the alkanes and alkanols. The sterol β-sitosterol was massive with 6.1 μg/g; Δ^5avenasterol was present in about half of its quantity. Stigmasterol is abundant as well. Minor sterols were poriferasta-7,25-dienol; cycloeucalenol, cycloartenol or oleana-11,13(18)-diene; cyclolaudenol or 24-methylene cycloartenol; campesterol; an unidentified stigmasterol relative; another unidentified compound; and a trace amount of cholesterol. It is likely that the cholesterol originated from the plant remains. This is remarkably similar to the sterol profile of fine-ware fragment 5486, but these two sherds are not from the same vessel. Sample 12461 is a

flared, medium-coarse rim that belonged to a constricted-neck vessel, while 5486 is a fine ware sauceboat. Both were slipped with Urfirnis. The only sterol from 12461 not readily present in 5486 was the unidentified compound that elutes right before stigmasterol.

Sample 2991 is a pedestaled base of medium-coarse fabric. It yielded 57.3 μg/g of lipid residue, of which 6.4 μg/g can be assigned to the sterol class. This is roughly half of the sterol content of 12461, yet it contained the exact same major and minor sterols. Sample 2991 contained a significant amount of β-sitosterol and Δ^5 avenasterol. Minor sterols were stigmasterol, campesterol, a relative of stigmasterol, and poriferasta-7, 25-dienol. Also found were the two compounds with multiple possible identifications: cycloartenol, cycloeucalenol, or oleana-11, 13(18)-diene and 24-methylenecycloartanol or cyclolaudenol. It also had retene, which is indicative of pitch (Robinson et al. 1987:641). This sample did not have an extensive range of fatty acids. The fatty acids of 2991 were mostly comprised of $C_{8:0}$ and even saturated fatty acids from $C_{12:0}$-$C_{18:0}$. Surprisingly, over a third of the fatty acid portion was unsaturated fatty acids, specifically $C_{16:1}$, $C_{18:1}$, and $C_{18:2}$. Fatty acids $C_{16:1}$ and $C_{18:2}$ could have been plant- or fish-derived. The presence of $C_{9:0}$ indicates the breakdown of even more $C_{18:1}$ (Mills and White 1994:40). There are signs of microbial breakdown with odd-chain fatty acids. Some saturated MAGs are present and trace DAGs. A range of alkanes with minor branching and some unsaturation were identified. Alkane 22 was the most abundant. Alkanols, however, were much more limited in range from 20-26 even and were present in low amounts. There were minor quantities of secondary alcohols and branched alkanols.

Although cholesterol was found, sample 2991 appears heavily plant-based. The small amount of cholesterol could be attributed to the trace amounts present in plants. In

the neutral portion, the cholesterol was present in 0.02 μg/g and in the TLE, cholesterol was 0.13 μg/g. For scale, β-sitosterol is 26 more times abundant than cholesterol in the TLE and 127 times more abundant than cholesterol in the neutral. Sample 2991 has more MAGs, more FA, and overall a greater abundance, but it is possible that 2991 and 12461 actually belong to the same vessel.

Summary

Here I have presented the results of the organic residue analysis of Ayia Triada pottery. I began with a data overview, where I discussed contamination issues, quantification, preservation, and some ubiquitous compounds. I then explained a series of lipid distribution studies I conducted by sampling sets of sherds from vessels. I followed with a summary of residue results by fabric and vessel type. I ended with a discussion of the evidence for the identification of beeswax in sherds and several high yield residues. Even though the substances cannot in many cases be delineated further than plants and animals, it is clear that the vessels and sherds analyzed in this study displayed a range of substances.

CHAPTER FIVE
DISCUSSION

The results reported in the last chapter can be used to better understand food consumption practices at Ayia Triada Cave. Four samples were considered empty and two samples were indeterminate, because either they were too degraded or contained no diagnostic compounds. The remaining 59 samples contained solely plant-based residues, animal residues, or complex mixtures of compounds. Here I examine these results in context of the hypotheses I presented in Chapter 1.

Food Remains in Serving and Storage Vessels

I hypothesized that the plants identified in the burnt organic layer were served or stored in the vessels deposited in the cave. I would expect to find either the biomarkers for these plants in the serving vessels as mixtures or as single sources, or I would expect to find that the storage jars have negligible quantities, which would suggest they held dry goods. Mavridis and Tankosić (2016a) suggested that the short, squat storage jars were used to transport the plant foods into the cave (Figure 5.1B). Analysis of the macrobotanical remains is not yet complete, but the limited range of plants that have been reported so far, i.e. figs, peas, lentils, and grains (Mavridis and Tankosić 2009b,2012,2016a), can be investigated in the residue data. The problem generally with plants is that they contain significantly less lipid content than animal resources (Reber and Evershed 2006:114). Cereals and legumes have proven difficult to detect with

Figure 5.1 Storage jar types found in Ayia Triada Cave

residue analysis and are likely underrepresented in organic residue results (Colonese et al. 2017; Hammann and Cramp 2018; Reber 2017; Reber and Evershed 2004). Lipid profiles, including known or potential biomarkers, and pitfalls for identification are discussed below for each of the plants reported in the cave so far.

There are numerous compounds found in fresh figs, *Ficus carica.* Figs have the ubiquitous phytosterols, β-sitosterol, stigmasterol, and campesterol, but also rarer psi-taraxasterol and fucosterol in addition to a series of triterpenes: lupeol, parkeol, and α- and β-amyrin (Jeong and Lachance 2001:280; Ribechini et al. 2011:3917). Figs also contain isovanillic acid and m-coumaric acid (Ribechini et al. 2011:3917). Most of the fatty acid profile (84%) is comprised of unsaturated fatty acids, $C_{18:3}$, $C_{18:2}$, and $C_{18:1}$, in order of decreasing abundance; fatty acids $C_{14:0}$, $C_{16:0}$, and $C_{18:0}$ are the only saturated fatty acids found in figs (Jeong and Lachance 2001:281). The most abundant biomarker compounds, and therefore those most likely to preserve in lipid residues would be β-sitosterol, psi-taraxasterol, and α-amyrin.

We do have some precedent for which compounds survive archaeologically. A single dried fig from eleventh century AD Islamic site Zaragoza in Spain was subjected to GC/MS; the only biomarkers that remained were β-sitosterol and α-amyrin (Ribechini et al. 2011). Unfortunately, the former sterol is very common in the plant world. The latter compound is less common, but still could not definitively identify figs without some of the other biomarkers discussed above. Not surprisingly, there was an almost complete depletion of the unsaturated fatty acids in the fig sample (Ribechini et al. 2011:3920). To my knowledge, figs have not yet been detected in any absorbed or visible residue analysis study. The extent to which the lipids found in figs survive in residues is

not known. Although many of the serving vessels contained β-sitosterol and unsaturated FA, I have not identified psi-taraxasterol or α-amyrin in any of the serving vessels or other sherds in this study.

Legumes, such as the lentils and peas found in the Cave, are rich in protein and phytosterols, but low in fat content (Geil and Anderson 1994; Ryan et al. 2007). Lentils and peas have 1.5 g/100 g of total fat, most of which consists of $C_{18:2}$, $C_{18:1}$, $C_{16:0}$, and $C_{18:3}$, in decreasing order (Ryan et al. 2007:89). They generally suffer from a lack of specific biomarkers. Lentils and peas contain the main plant phytosterols, β-sitosterol, campesterol, and stigmasterol and the less common, but no more diagnostic Δ^5 avenasterol. This is also true for broad beans, chickpeas, and most other legumes; the main difference between various legumes is the relative abundance of these sterols (Kalogeropoulos et al. 2010:685; Ryan et al. 2007). Very small amounts of cholesterol are also found (Kalogeropoulos et al. 2010:685). In all of the common pulses, β-sitosterol is the most common sterol (Weihrauch and Gardner 1978:43). In lentils, campesterol, stigmasterol, and Δ^5 avenasterol are in roughly equivalent amounts (Kalogeropoulos et al. 2010:685). In fava beans, chickpeas, and peas, campesterol is more abundant than Δ^5 avenasterol, which is more abundant than stigmasterol (Kalogeropoulos et al. 2010:685). Although sterol content alone could not identify legumes from many other plants, if there were other evidence to support the presence of legumes, the relative sterol content could possibly be useful for differentiation. Tocopherols in α, β, δ, and γ forms are also present in lentils, with the coelute of β and γ being most abundant (Kalogeropoulos et al. 2010:687). For peas, α-tocopherol is present in significant amounts, nearly two times as much as the coeluted β/γ tocopherol (Ryan et al. 2007:89). Lentils and peas also have a

series of phenolic acids, including o- and p-coumaric acid and vanillic acid (Kalogeropoulos et al. 2010:685). Lentils yield p-coumaric in highest quantity, while peas yield o-coumaric and ferulic acid in highest quantities (Kalogeropoulos et al. 2010:688). Once again, these compounds are present in many legumes, but vary in relative abundance. Three triterpenic acids if found as a suite (oleanolic acid, ursolic acid, and maslinic acid) could differentiate lentils and chickpeas from broad beans and peas at least (Kalogeropoulos et al. 2010:688, Table 5). However, this exact trio of compounds is also found in the Lamiaceae family, which includes rosemary and sage, and in olive oil, while individually, these are found in many more plants (Boskou 2002:256; Razborsek et al. 2008). Unfortunately, these are not robust biomarkers either.

It appears that legumes are difficult to unambiguously detect via biomarkers. Many of the main sterols that are likely to survive and that have indeed survived in the Ayia Triada serving vessels could not differentiate between legumes and other plants. There is no evidence yet for any of the minor compounds found in legumes in the serving vessels. Kalogeropoulos and colleagues (2010:686-87) reported that the sterol, tocopherol, and polyphenols were much lower for cooked than dried legumes. Most of the reporting of compounds outlined above is based on amounts found in dried legumes. That means that for serving vessels, like the ones here, where legumes would have been cooked, these minor compounds would likely be undetectable.

The identification of cereals suffers from similar issues as legumes and may be even more difficult to detect. Cereals have similar low fat levels and much lower tocopherol and sterol content than legumes (Ryan et al. 2007: 87-88). Barley and wheat have been initially identified in the plant assemblage from Ayia Triada (Mavridis and

Tankosić 2012; 2016a:223). Barley and wheat contain β-sitosterol as the predominant sterol, with minor sterols stigmasterol and campesterol (Ryan et al. 2007:88). There are some unusual esters of odd chain fatty acids between $C_{31:0}$-$C_{37:0}$ and secondary alcohols from C11-C17 in wheat, barley, and rye (Bianchi 1995:188). Saponification would break these into compounds that would show up in the FA and N portions. It is often difficult to identify secondary alcohols to their carbon chain length, because the molecular ion is usually not present in the mass spectrum. Barley contains an ester of 7-oxopentadecan-2-ol with $C_{18:0}$, $C_{20:0}$, and $C_{22:0}$ (Bianchi 1995:188). Diesters of short diols are found in wheat and rye (Tulloch 1976 and references therein). Hydroxy β-diketones have been found in the Poaceae (formerly Gramineae) family to which wheat and barley belong (Tulloch 1976:247). More research needs to be conducted on these possible biomarkers.

Interestingly, none of these potential biomarkers were recovered in Hammann and Cramp's (2018) experiments on the cooking of rye, barley, and spelt. They did uncover a new biomarker sequence in cereals, a series of alkylresorcinols. They found very small amounts of these compounds using an enrichment protocol and very sensitive GC with quadrupole-time-of-flight mass spectrometry after successive boiling episodes of cereals (Hammann and Cramp 2018:77-78). These compounds were confirmed archaeologically in two pottery sherds of unknown type, but the amounts recovered were miniscule (< 1 ng/g). Alkylresorcinols are not as stable as even sterols and are more susceptible to microbial degradation (Hammann and Cramp 2018:79). Uptake of these compounds was increased when cooked with other substances, in particular meat. To detect these compounds, a targeted extraction protocol with more sensitive GC/MS instrumentation must be employed.

No evidence has emerged for figs, cereals, or legumes in the serving vessels, although this discussion has aimed to show how complex it is to prove, given the lack of highly diagnostic biomarkers. Dry storage of these plant foods is a possibility, maybe in the short, squat jars. If so, the residues would probably display lipid quantities of less than 5 µg/g (Eleanora Reber, personal communication 2018). In all but one of these short, squat jar samples, lipids were recovered in quantities well above 100 µg/g, as mixtures of pine resin, animal, and plant materials. Therefore, there is no evidence to suggest that the dried plants found in the cave were solely stored on these jars. Better methods for detecting dry goods in storage jars would be via phytoliths, starch grain, or protein analysis.

Liquid Storage

I hypothesized that two jar types, referred to here as the ovoid and bulbous types, held either single commodity liquids or alcoholic beverages. The shape of these vessels suggests the storage of liquids. Large, sturdy strap handles and a constricted neck support this interpretation for the tall, ovoid type (Figure 5.1A). I analyzed sherds from two of the ovoid-type jars. A constricted neck, podes that could be used to attach a covering, and a possible drainage hole on the bulbous type suggest its association with liquids (Figure 5.1C). The hole appears to have been intentionally pierced prior to the firing of the vessel, because a clear finger wipe encircling the hole is evident in the clay. Only one jar of this type was analyzed. Good candidates for liquid contents of these jars would be olive or another edible oil; or wine, beer, or another alcoholic beverage. It is also possible that these types merely held water, which would appear as ‘empty’ residues.

Vegetable Oils

Olive oil can be difficult to detect because of its lipid profile. Over 98% of olive oil is comprised of fatty acids and acylglycerides, most of which are unsaturated TAGs that are very prone to oxidation and microbial breakdown (Gunstone and Harwood 2007:47-48; Servili et al. 2004:114). After only 95 days, experimental evidence has shown that TAGs in olive oil are greatly reduced and the overall lipid quantity is decreased by more than 90% (Dudd et al. 1998: 1349-1350). Minor amounts of saturated fatty acids, $C_{14:0}$, $C_{15:0}$, $C_{20:0}$ and branched $C_{15:0}$ and $C_{17:0}$, emerge throughout the degradation process (Dudd et al. 1998:1350). Olive oil yields high levels of oleic acid ($C_{18:1}$) (Gunstone and Harwood 2007:47). In a study of 78 Greek olive oils, the mean relative abundance of oleic acid was 76.9% (Boskou 2002:248). The second most common fatty acid is $C_{16:0}$, albeit a far second, and minor fatty acids are $C_{18:2}$, $C_{18:0}$, $C_{16:1}$, and $C_{18:3}$ (Gunstone and Harwood 2007:47-48).

The other 2% of olive oil is comprised of 230 minor compounds, including sterols, triterpenoid alcohols, tocopherols, and hydrophobic phenolics (Ramírez-Tortosa et al. 2006:56; Servili et al. 2004:114). The latter compound type, which includes a benzoic and cinnamic series among other compounds, is not found in other vegetable oils (Boskou 2006:52-83). The main sterols are the same as in legumes. Some of the minor sterols are cholesterol, obtusifoliol, cycloeucalenol, gramisterol, citrostadienol, 24-methylene-cycloartenol, and β-amyrin (Boskou 2002:253-254). Squalene and α-tocopherol are relatively abundant and comprise 40-50% of the neutral component (Gunstone and Harwood 2007:48; Ramírez-Tortosa et al. 2006:55-56). Olive oil has an unusually high content of squalene (Liu et al. 1976:38). Two triterpenoid alcohols present

in olive oil are ethythrodiol and uvaol (Boskou 2002:255; Ramírez-Tortosa et al. 2006:53). Many of these compounds discussed are present in such trace amounts that it seems unlikely that they would survive archaeologically. In the case of squalene, it rapidly breaks down in a matter of months because of its high level of unsaturation (Archer et al. 2005). There is no one compound that can both unequivocally identify olive oil and that is likely to survive. Rather, a suite of compounds is used to suggest degraded olive oil: the main plant sterols; unsaturated TAGs, DAGs or MAGs; high levels of $C_{18:1}$; $C_{9:0}$ and to a lesser extent $C_{8:0}$ and $C_{10:0}$; and C9 and C7 diacids (Gunstone and Harwood 2007; Mills and White 1994; Regert et al. 1998).

TAGs were not recovered from any Ayia Triada samples, so one of the main indicators of olive oil is not preserved in any vessel. MAGs or DAGs with oleic acid components could indicate the presence of their original TAG counterparts, however. The samples that belonged to the ovoid jars were 5436, 7684, and 7063. Samples 5436 and 7684, which were from the same jar, had very little residue. Sample 7684 had < 5 μg/g. Although 5436 and 7063 had unsaturated $C_{18:1}$, it was present in very small amounts (< 1 μg/g). None of the acylglycerides had $C_{18:1}$ moieties. There were no diacids and little $C_{9:0}$ in 5436. Moving to the one bulbous jar, sample 8020 did not contain $C_{18:1}$, unsaturated acylglycerides, or diacids, and only a small quantity of $C_{9:0}$. There was over 100 ug/g of lipid residue. It seems unlikely that no $C_{18:1}$ would survive in this otherwise well-preserved residue, especially if substantial levels of this fatty acid were once present. Its pair, sample 4469, had 1.0 ug/g of $C_{18:1}$ and scarce $C_{9:0}$, but the $C_{18:1}$ only makes up 0.88% of the residue. There were no diacids or $C_{18:1}$ moieties in the acylglycerides. The

sample displayed good preservation with 118.6 ug/g total residue. None of these storage jars contained enough evidence to suggest the presence of olive oil.

Other vegetable oils were likely used in the Early Bronze Age, *Lallemantia* and linseed oils. Linseed oil, the oil from flax seed, is comprised of 43-47% $C_{18:3}$ and roughly half that amount each of $C_{18:2}$ and $C_{18:1}$ (Gunstone and Harwood 2007:46; Ryan et al. 2007:89). It is comprised predominantly of γ-tocopherol, β-sitosterol, campesterol, stigmasterol, and Δ^5 avenasterol (Kochhar 2002). Minor sterols in linseed oil are poriferasta-7,25-dienol, cycloeucalenol, cycloartenol, 24-methylene-cycloartenol, obtusifoliol, 24-ethyllophenol, and brassicasterol (Kornfeldt and Croon 1981). While most seed oils have trace amounts of cholesterol, linseed oil has appreciable amounts of this sterol usually only associated with animal products (Weihrauch and Gardner 1978:41). *Lallemantia* oil is very similar to flax in its main sterol content, TAG distribution, and tocopherols (Zlatanov et al. 2012:1397-1398). It has higher $C_{18:3}$ levels than flax, however (Zlatanov et al. 2012:1398). This oil is a minor one in modern times and therefore, has not been as extensively researched as olive oil and to a lesser degree linseed oil. The minor compounds of *Lallemantia* oil have not been identified yet, but they may be similar to those of found in linseed oil. High $C_{18:3}$ levels could indicate the presence of either of these oils. However, $C_{18:3}$ is even more likely to degrade than $C_{18:1}$, because of the two additional double bonds. None of these jars, or any samples in this study, recovered $C_{18:3}$.

As a side note, it seems that residue analysts may be too quick to equate cholesterol unanimously with animal products, regardless of the amount present and range of other sterols. More discretion may be needed, especially in the presence of

abundant plant sterols. In this study, I have more carefully considered residues that have plant sterols that are both abundant and present in significantly higher quantities than cholesterol. I have only assigned a residue containing cholesterol as solely plant-based when the phytosterols comprised 96% or more of the total sterol content in the TLE. Quantified in a slightly different way, phytosterols are present in amounts that are 27 or more times the amount of cholesterol in these samples.

Compelling evidence for vegetable oils may be lacking in the purported liquid storage jars, of which only a few were tested, but it is possible that some of the other storage jars and sherds of unknown type held vegetable oils. Samples 8096 and 4972 belonged to the same short, squat jar with a large opening. Sample 8096 contained $C_{8:0}$, $C_{9:0}$, and $C_{10:0}$ in roughly equal, albeit small, amounts. These three fatty acids are the result of $C_{18:1}$ breakdown (Mills and White 1994:40). It contained an appreciable amount of unsaturated fatty acids, $C_{16:1}$, $C_{18:1}$, $C_{14:1}$ and $C_{17:1}$, in order of decreasing quantity. The relatively abundant $C_{16:1}$ likely originates from the hydrolysis of the $C_{16:1}$ wax esters in this sample. Therefore, $C_{18:1}$ is the second highest amount. Additionally, more $C_{18:1}$ was bound in a MAG. Sample 4972 had similar amounts of free $C_{18:1}$ and MAG 18:1 as sample 8096. It contained only $C_{9:0}$ and $C_{8:0}$. Although C9 and C7 diacids were not present in this sample, the short chain fatty acids show breakdown of $C_{18:1}$. Without some of the minor compounds, we cannot definitively assign this residue as olive oil, as it could just as likely be linseed or *Lallemantia* oil. Sample 8096 contained the appropriate main sterols for these three oils, β-sitosterol, Δ^5 avenasterol, and stigmasterol, while 4972 contained only germanicol and stigmastane. Caution should be exercised here, because of the other lipids present in this sample. This residue might merely originate from a very

oily plant, because significant quantities of wax esters were found in sample 8096 with $C_{12:0}$-$C_{14:0}$, $C_{16:0}$, and $C_{16:1}$ fatty acid chains. Wax esters are not found in oils. Also, these samples both contained cholesterol and 7-ketocholesterol and 8096 had a third cholesterol derivative, cholesta-3,5-dien-7-one. This could reflect a mixture of vegetable oil or other plant-derived material with meat. However, it could also reflect the application of animal lard as a sealant to the vessel. The possibility of animal product sealants will be discussed later in this section.

Three other samples that possibly contain vegetable oil are 2062, 2991, and 3133 A, based on the same fatty acid and diacid criteria. Sample 2062 is the only from these three that is from a known vessel; it is a yellow-mottled ware sauceboat. It had relatively high amounts of unsaturated fatty acids, 11.5 µg/g, which comprises 24% of the fatty acids found in the TLE. Specifically, it contained $C_{16:1}$ in two isomers, $C_{18:1}$, and $C_{18:2}$. $C_{18:1}$ is the most abundant of the four and it was likely even more abundant originally, as indicated by $C_{8:0}$-$C_{10:0}$. This sauceboat also had the suite of general plant sterols that would be found in vegetable oils and abundant cholesterol, which is certainly more perplexing. This sample will be discussed further in the Sauceboat section.

Samples 2991 and 3133 A appear to be the most likely candidates to contain vegetable oils from the unknown sherds, although the latter is the more tentative. Sample 2991 contained $C_{16:1}$, $C_{18:1}$, and $C_{18:2}$. $C_{18:2}$ was the most abundant of the unsaturated FA. Nevertheless, this sample contained $C_{9:0}$ and $C_{8:0}$ indicating more $C_{18:1}$ was once present. It contained the major plant sterols found in oil, β-sitosterol, Δ^5 avenasterol, campesterol, and stigmasterol. It also contained minor amounts of three unusual sterols reported in the Results chapter that are all found in linseed, poppy, sesame, and wheat germ oils.

Sample 3133 A contained 1µg/g of $C_{18:1}$, small amounts of $C_{8:0}$-$C_{10:0}$, and C9 diacid, but no unsaturated acylglycerides or sterols. It contained many of the compounds that would be expected in degraded vegetable oils, but in miniscule amounts considering the copious amount of residue recovered, 1,001.4 µg/g. This was also one of the best-preserved beeswax residues from this assemblage. Such a strong beeswax signal could be masking other compounds in the TLE. If beeswax was used as a sealant in this vessel, which is only one possibility, it could have prevented any substantial absorption of the oil into the vessel walls. It is equally plausible that what we are seeing is not oil in a beeswax-sealed vessel, but a mixture of commodities or the additive effects of different resources stored or served independently throughout this vessel's lifetime.

Alcoholic Beverages

The topic of how to identify alcoholic beverages in organic residues is still rife with debate (Evershed 2008a; Michel, McGovern, and Badler 1993; Steele 2013). Alcoholic beverages contain mostly water with sugar and alcohols, which are themselves easily dissolved in water and are prone to disappearance after burial (Steele 2013:95). The biomarkers for wine have often been touted as tartaric acid and, for red wine only, syringic acid (Guasch-Jané et al. 2004; McGovern 2007, 2009; Michel, McGovern, and Badler 1993). However, the presence of tartaric acid cannot be used to differentiate between grape juice and fermented grape juice (Michel, McGovern, and Badler 1993:411). Furthermore, although tartaric acid is found in high quantities in grapes, it is not exclusive to grapes; it is also found in dates and pomegranates, for example (Michel, McGovern, and Badler 1993:408; Singleton 1996). Syringic acid is released after alkaline

treatment of malvidin, the color-giving compound found in red wine (Guasch-Jané et al. 2004:1672). It is likely found in fewer plants than tartaric acid, but it is found in pomegranate, whortleberry, red clover, high mallow, and olive oil (Barnard et al. 2011:978; Boskou 2002:258). Pecci and colleagues (2013:114) found "tartaric acid, together with malic, succinic, fumaric, and citric acids" in what they believed to be wine processing vats and these compounds were confirmed experimentally; these could be a potential suite of biomarkers for wine.

Neither tartaric acid nor syringic acid were found in the liquid storage jar samples, nor were they identified in any other vessel or sherd sampled from Ayia Triada. Surprisingly, even GC/MS may not be able to identify tartaric acid and syringic acid; a more sensitive method, such as high-performance liquid chromatography and mass spectrometry in tandem mode, may be required to identify these compounds in complex mixtures, since they often survive only in minute amounts (Guasch-Jané et al. 2004; Stern et al. 2008). Guasch-Jané and colleagues (2004) were able to extract tartaric acid and syringic acid from very small sample sizes of Egyptian amphorae using this method; this was the first time syringic acid had been found archaeologically in connection to wine. Tartaric acid is particularly water soluble and may only preserve in very dry climates (Barnard et al. 2011:978; Michel, McGovern, and Badler 1993:408). More secure wine identifications are made in conjunction with archaeological and palaeoethnobotanical evidence, as in Barnard and colleagues' (2011) study of a wine installation or with epigraphic evidence, as in Guasch-Jané and colleagues' (2004) study of wine-labeled amphorae.

Beer too is difficult to detect, and no reliable biomarkers have been identified. Ergosterol could provide a biomolecular signature for fermentation in the beer, but this sterol is not only found in beer and can merely indicate fungal breakdown of any substance (Isaakson et al. 2010; Steele 2013:96). "Beerstone" has been suggested as a possible biomarker for barley beer, which is identified mostly as calcium oxalate, but it has also been detected in some plants (Michel, McGovern, and Badler 1993). Unfortunately, it too is water soluble and the likelihood of it being found in a non-arid climate is low. Regardless, neither of these supposed biomarkers have been found in the Ayia Triada samples.

Water

With no evidence for fermented beverages or vegetable oils in the ovoid or bulbous type jars, it is possible that they could have been empty or held water. Both uses would appear the same in terms of residues, or lack thereof. All but one of these samples had appreciable amounts of lipids preserved in them. The tall, ovoid jars generally had less lipids with the maximum lipid content of 25.4 µg/g; the bulbous type had a maximum of 163 µg/g. The bulbous type had beeswax and pine resin, while the ovoid jars had neither. Otherwise, both jar types had evidence for animal products and more than one plant, including epicuticular wax components. These jars were certainly not empty. Although we cannot discount that may have been used as water jars at some point, they would have clearly been reused for other substances that left chemical signatures within the vessel walls.

Sealants

Another line of evidence for liquid storage is sealants applied to the vessel interior as a post-firing treatment. Possibilities for sealants are beeswax, various resins, scalded milk, fruit juices, or even animal fats, to name a few (Arnold 1985:140; Charters et al. 1995; Rice 1987:163; Stimmell and Stromberg 1985). Surprisingly, only one of the suggested liquid jar types has been tentatively identified as having beeswax, the bulbous-type vessel. Beeswax could have been used to reduce liquid absorption into the clay matrix (Charters et al. 1995; Heron et al. 1994). The practice of applying beeswax to seal vessels is known ethnographically. Spathari-Begliti (1992:149) reported beeswax sealants used by Greek potters from Siphnos. Kelley and Orr (1976) described the process of Ecuadorian potters who coated storage vessels with beeswax and tar immediately after pulling them from the fire to waterproof the interior.

Beeswax was not only identified in the bulbous type. It was found in two of the short and squat storage jars, in all representing 27% of storage jar samples. Beeswax was also found in five other sherds of unknown vessel type. It was not, however, found in any of the bowls, sauceboats, pyxis, or the open, handled vessel.

It is important to point out that other possibilities exist for the presence of beeswax in the storage jars, besides its use as a sealant. Beeswax could indicate that honey was added as an ingredient or a flavoring, since raw honey can contain bits of beeswax that had not been filtered out. It seems that if it was only a flavoring or ingredient, it would have been more dilute in the original substance, and therefore would not have produced residues that were overwhelmingly comprised of beeswax components as seen here. Another possibility is that honey was the sole resource stored in the vessel.

The presence of other compounds could be the remnants of previous substances. These jars seem on the larger size for storing a scarcer resource like honey, especially the deeper, bulbous jar, but this is still a possibility. The storage jars also could have held beeswax itself that was being collected for later use. We would expect no other compounds to be found in the residue, unless again the jar was reused for different resources at some point. Of these four possibilities, it seems least likely to have been used as an ingredient or flavoring in these storage jars, because of the significant portion of the residue that the beeswax compounds comprise. This is very different from the pine resin remnants in the Ayia Triada sherds, for example, that are never abundant, being recovered in quantities of less than 1 μg/g.

This is not the first time beeswax has been found in Greece, nor is it the earliest. The earliest confirmation of beeswax is in the so-called 'cheese pots' from the Northern Greek sites of Limenaria and Dikili Tash, dating to the Middle and Late Neolithic I, respectively (Decavallas 2007; Regert et al. 2001). One sherd was tested from each site from these perforated vessels. Decavallas (2007:154) argued that the vessel was a beeswax-fueled lamp, a beehive, or a honey strainer. The holes preclude any possible storage function and would argue against the use of beeswax as a sealant for this vessel (Decavallas 2007:154). The next period for which beeswax has been discovered is the Middle Bronze Age. Beeswax has been found at Chamalevri dating to the Middle Minoan I A period in a channel used to route liquids in a workshop and a miniature vial that was possibly used as a measure (Tzedakis and Martlew 1999:51). It has also been found at Akrotiri in the Middle to Late Bronze Age in cooking pot sherds (Roumpou et al. 2003:34). Beeswax was discovered in lamps dating to Late Minoan I period at Mochlos,

in which beeswax was possibly used as fuel (Evershed et al. 1997). It has also been found in Thebes in a cooking jar and Mycenae in an angular bowl (Tzedakis and Martlew 1999:51). The two most relevant findings for this study were beeswax identified in Late Bronze Age storage containers from the Thessaloniki Toumba (Margomenou and Roumpou 2014; Roumpou et al. 2003) and beeswax in five pithoi dating to the Late Minoan IB period of House 2 of Galatas Pediados (Christakis 2005:52; Christakis and Rethemiotakis 2011:180). In both cases, it was argued that beeswax was used as a sealant, based solely on its finding in storage jars. Beeswax has also been identified in later Historic periods, such as in combed pottery from the Hellenistic and Roman periods (Evershed et al. 2003).

There is a clear lacuna in our knowledge for the use of beeswax in Greece between the Neolithic and the Middle Bronze Age. The present study stands out as the only EBA evidence discovered thus far for beeswax. In addition, it is the earliest finding of beeswax in storage jars. If the residues from the storage jars reflect beeswax as a sealant, this would push back the earliest dates for the practice nearly 1500 years. If it reflects honey or beeswax storage, this would be the first finding in the EBA as well. It is impossible to gauge how widespread beeswax or honey use was in this period until the remaining Ayia Triada samples are analyzed and more samples are analyzed from other sites. There is at least initial evidence that suggests that it may be a local idiosyncrasy of the inhabitants of Karystia. There has been one other study to my knowledge that analyzed storage jar residues dating to the EBA II period. In Psaraki and colleagues' (2013) study of the mainland site of Thebes, 10 storage jar sherds from four different types were analyzed via GC/MS. Beeswax was not found in any of them, nor was it

found in any of the other storage vessels (Psaraki et al. 2013:94-98). It is worth mentioning that there seems to be a sampling bias against testing storage jars for organic residues (Roumpou et al. 2003). It is often assumed that no lipids would be preserved since heat was not applied to the vessel and because of the dry storage uptake issue. The fact that no other evidence for beeswax in storage jars has emerged for the EBA could merely be a result of this; the practice may be discovered elsewhere in the future. With broader regional evidence, we may be able to narrow down how beeswax or honey was being used.

Two other substances could have been utilized on the liquid storage pots as sealants, pine resin or animal fat, given residue results. One of the liquid storage jars had evidence for pine resin, the bulbous jar 8020/4469, while the two ovoid jars did not. However, the minute amount of DHA or its oxidation products found in 8020 and 4469 within an overall abundant residue suggests that it was used as an ingredient, instead of a sealant. A stronger signal would be expected if pine resin alone coated the entire interior of the vessel. It is entirely possible that the cholesterol found in the jars represents an animal fat used as a sealant. Only trace amounts of cholesterol were found in the ovoid type jars. The quantities were not that much greater in the bulbous type, between 0.1 μg/g and 0.4 μg/g. It is possible that the beeswax in the bulbous jar was mixed with some animal fat and pine resin in a multi-ingredient sealing solution. The addition of animal fat to beeswax could help to stretch this scarcer resource farther (Evershed et al. 1997); the pine resin may have added some desired quality. Mixtures of sealants are known ethnographically, but it is challenging to tease out a multi-ingredient sealant from a single

sealant in a jar holding multiple contents. This discussion underscores how difficult it is to disentangle individual resource signatures from mixed residues.

What is very clear is that the ovoid type had no obvious sealants and little lipid content, while the bulbous type could have had a beeswax sealant and contained substantial amounts of lipids. The latter would be a well-prepared vessel in which to store liquids and the curious hole may in fact be a drainage hole. Although no possible sealants were found in the ovoid type, the lipid distribution found within it suggests a lipid-poor liquid, but the identity of this liquid cannot be determined.

Vessel Types

I hypothesized that there is homogeneity of use within the pottery types analyzed here. I sampled from seven bowls, one pyxis, 16 jars, two sauceboats, and one open, handled vessel. I expected to see relatively similar compounds and quantities within categories and differences between categories, reflecting unique uses of each vessel type.

The pyxis (129) and the open, handled vessel were the only ones tested of their respective types. The pyxis contained a mixture of animal and plant resources, likely from only one plant. The open, handled vessel (11309/3482) was possibly a cooking jar, based on the synthesis of ketones. It contained a degraded mixture of multiple plants, one of which was pine resin, and meat. Pine resin was not found in both sherds tested from this vessel, only in the body sherd, and it was present in a small quantity (0.4 μg/g). This suggests that pine resin was used as an ingredient rather than a sealant applied to this vessel. It is not possible to draw any broad conclusions about these vessel types, since only one sample was tested each.

Two sauceboats were sampled for residue analysis and both had recoverable residues. One of the sauceboats, 5486/7370, had compelling evidence for an exclusively plant-based residue, while the other sauceboat contained more than one plant and animal resources. These important vessels will be discussed further in the last section.

Most of the seven bowls had evidence for a mixture of animal and plant foodstuffs without any pine resin or beeswax. Sample 5510, the only coarse-ware bowl, was the one exception to this; it originally contained only a small range of plants. The three rims all had the lowest amount of lipids with less than 50 μg/g, which confirms that these were not cooking vessels or at least not vessels in which foods were boiled. One of these rims belonged to a bowl that was sampled on the body as well. The spatial distribution with more lipids towards the base upholds the presupposition that the bowl was not subjected to high heat.

It initially appeared that the rest of the bowls were rather homogenous, but upon closer inspection, subtle differences were observed in their lipid profiles. The signature for animal resources was the same across the bowls with a cholesterol biomarker. However, the plant content was more limited in some bowls than others. Two bowls (8428 and 15787/3040) showed evidence for only one to two plants with a small range of alkanols. The other three bowls, 8092, 7599, and 5175, had a wider range of alkanols from C20 to C30, C32, or C34, indicating that multiple plants were served in the vessels. Samples 8092 and 7599 were shallow bowls, while 5175 was a deep bowl of medium size with a larger diameter. Sample 5175 stands out from all bowls with a high residue yield and more varied contents. It contained a more complex residue with substantial secondary alcohols, wax esters, more unsaturated fatty acids, o-coumaric acid, and the

largest amount of labdane in any sample, although the latter might represent post-excavation contamination. Bowl 5175 could have been the bowl from which the foodstuffs found in 8092 and 7599 were served or it could just have been used more intensively throughout its lifetime. The two smaller bowls were found together in a feature with human bones, while bowl 5175 was found in Level 5b, which was in a different trench. Although the exact plants are not known, it is clear that some bowls once contained more diverse plant components than others.

I sampled from several types of reconstructed storage jars. One type is a short and squat storage pot with an everted lip and short neck (Mavridis and Tankosić 2016a:225). I collected six samples from this type, which represent four jars (3038/3292, 4972/8096, 7056, and 5025). Another type is a taller, ovoid shape with large strap handles, a constricted neck, and yellow-mottled surface decoration. I collected three samples from two of these ovoid jars (5436/7684 and 7063). A third jar type is a bulbous-shaped jar with a constricted neck, small podes on the upper body, and a curious hole. Only one jar like this was sampled with two body sherds, 8020 and 4469. The previous two jar types were discussed in detail in the Liquid Storage section. A fourth type is a pithos with a flared neck, a decorative rope band at the juncture between neck and body, and podes on the exterior (Figure 5.1D). The jar did not have substantial residues and had no evidence for beeswax or pine resin. Three sherds were sampled from it that did not directly mend to the larger reconstructed piece. As discussed in the Results chapter, one sherd does not appear to belong based on its lipid content. Two other sherds, 1686 and 8506, are from storage jars, but the type to which they belong cannot be determined.

All four short and squat jars had lipid profiles suggesting pine resin, plants, and animal products. However, two of these were tentatively identified as having beeswax as a sealant or as the vessel contents, while the other two had no evidence for beeswax. Since they are all the same type of storage jar and all had pine resin, it is tempting to explain this lack of beeswax on the grounds of differential preservation. The two jars with beeswax yielded residues between 160 µg/g and 433 µg/g, while the two without beeswax yielded between 45 µg/g and 105 µg/g of residue. Maybe the beeswax merely degraded faster or more thoroughly in these other jars. This could be the case for the sample that had the smallest residue quantity of all the squat jars, sample 5025. However, it seems unlikely to have such preservation differences on vessels from the same small site. Furthermore, jar 4972/8096 was well preserved and contained a different lipid profile than the rest. Sample 8096 had the highest quantity of wax esters found in any sherd, but they were not the wax esters specifically found in beeswax. Therefore, there is no reason to believe that beeswax would not have survived in this jar if originally present.

Another alternative that has not been discussed yet here is that beeswax was being used as an insecticide inside the vessel, instead of a waterproofing agent, possibly for a products known to attract pests, such as grain (Roumpou 2003:197). Christakis (2005:32) discussed that cereals, legumes, olive oil, and wine were stored in beeswax-sealed pithoi. It is unclear if he is referring to ethnographic observation or archaeological evidence. Regardless of whether we view beeswax as a sealant, an insecticide, or the stored good itself, its presence in only some and not all jars suggests specialized storage.

The vessels from which enough specimens were sampled to make fruitful comparisons are the jars and bowls. The presence of beeswax and pine resin in some

storage jars is a major difference. The pine resin is more challenging to determine if it was an ingredient or a sealant due to its small amount usually. It was found in two of the four storage jar types and not any bowls. Surprisingly, there is more heterogeneity within types than expected, which does suggest a range of foods or goods being served or stored in both vessel types.

Imported Versus Local Wares

I hypothesized that the imported wares contained different organic contents than the local wares, reflecting specialized usage of these types. These differences could be in the range of resources, the quantities found, or scarcity of the commodity. Imported wares comprise 17% (11 of 65) of the samples analyzed to date. Two types are deemed imports because of their fabric color, specifically the buff and grey fabrics. The other import is based on surface decoration, which is burnished with a yellow to pinkish slip. It is known as yellow-mottled ware and is typical of the mainland (Broodbank 2008:64-65).

All of the non-local fabric types are fine wares. The buff fabric sherd was the pyxis discussed above, sample 129, with degraded plant wax from more than one source and animal fats. The grey fabric wares, samples 7219, 7078, and 8156, had smaller amounts of residue (< 30 µg/g). It is possible that these postdate the EBA, but this cannot be confirmed until the pottery analysis is completed (Žarko Tankosić, personal communication 2018). Sample 8156 was empty. Sample 7219 had no cholesterol and was entirely plant-derived. Sample 7078 had a small amount of cholesterol, but was otherwise plant-based. There is nothing particularly remarkable about these residues. As a group, they seem to be somewhat less abundant than other residues, but generally display the

same range of substances. They might have been used less frequently than some of the other sherds, which accords well with them being fine wares.

The yellow-mottled wares are mostly storage jars with strap handles, samples 5436, 7684, and 7063, while two others are of unknown vessel type, samples 1686 and 8849. The yellow-mottled surface of jar 5436/7684 had been damaged by burning. These two samples also have the lowest amount of lipids from any of the yellow-mottled wares at 9 μg/g and <5 μg/g, which could be a result of the extensive burning observed in their associated stratigraphic layer, Level 4. Burnishing is still evident on these sherds, even though the yellow color has changed to grey/black. The only other yellow-mottled ware is a sauceboat (2062), which will be discussed in more detail in a later section. There was no evidence for beeswax in any of these sherds. Pine resin is present in only two of these wares, the sauceboat and an unidentified body sherd (8849). Besides the presence of pine resin in some, the yellow-mottled wares all apparently contained plants, including epicuticular wax from more than one plant, and animal resources. It is not possible to assign the residues any further. The fact that pine resin is also identified in local wares establishes that it is not a substance restricted for use only in imported types of pottery.

Surprisingly, these residues from the buff-fabric, grey-fabric, and yellow-mottled wares cannot be differentiated substantially from the local wares. It is easy to assume that imported pottery types held differentiated or unique contents, but this research shows that imported wares do not necessarily have to contain any specialized contents, per se. Owners of these vessels could have used imported wares for special situations, much like one would use the fancy china only for certain holidays or events, but generally serve the

same foodstuffs in them. This tests our assumptions of the meanings of the pottery groups we designate and the actual behaviors they may or may not reflect.

Handles

In this study, I tested the suggestion first put forth by Heron and Evershed (1993:256) that handles can be used to gauge soil contamination. I would expect low lipid amounts if the handle residues originated from soil contamination. The quantity and quality of residue recovered from handles 8141 and 1656 were considerably more extensive than that expected in soil (Heron, Evershed, and Goad 1991). Although the amounts found, 55.1 µg/g in 8141 and 135.17 µg/g in 1656, were alarming at first, the fact that lipid residues could absorb into handles during use can be reconciled on a few levels. On one level, substances on hands can easily be transferred to handles. The possibility of lipid transfer from human hands is the entire premise behind wearing gloves during residue sampling and extraction. One needs to only look at a white keyboard or light switch to see the grime that is transferred from human hands upon daily use. If a person was cooking and touching the vessel handle with food-soaked hands regularly, there is an even greater chance of lipid transfer. On another level, if the handle was located on a cooking pot used to boil foodstuffs or industrial substances, it is very likely that contents would boil over onto the exterior of the vessel and therefore, absorb into the handle. Handles are likely more porous than the interior of pot; it seems unlikely that they would have been sealed intentionally or indirectly with the first few uses of a new pot. If any substances did splash on them, they would absorb them more readily.

This transfer of lipids to cooking pot handles has been confirmed, at least ethnographically, in one case. Evershed (2008b:35) sampled the handle of a traditional Greek cooking dish from the last century and found a massive amount of lipid, 1.3 mg/g. This is significantly greater than the quantity found in the handles tested in this study, but it shows that there is precedent for lipid absorption beyond the interior. I would argue that these handles might have been attached to cooking pots too and that the residues represent a lifetime of spillovers from cooking episodes. Additionally, if the residues found in the handles were merely background soil contamination, one would not expect there to be any 'empty' sherds found in this study at all and this is clearly not the case. The fact that the residue from these handles quantitatively and qualitatively differed from each other is another clue that they are not the results of merely soil contamination.

The finding of beeswax in handle 1656 is curious. It is possible that the handle was coated with beeswax as a stylistic choice, since it would add a certain sheen. Or it is conceivable that it was merely transferred from the potter's hand if they were coating the vessel with beeswax. In that case it would have been an accidental addition. It also could have been a flavoring or ingredient of whatever substance was being cooked or processed in the vessel to which this handle belongs. Finally, if honey or beeswax was stored in the vessel in at least one usage, it could have splashed onto the handle. Once the vessel type is determined, if it is possible stylistically, this could shed light on the purpose of the beeswax.

There has been one other study to my knowledge that analyzed archaeological handle residues. Roumpou and colleagues (2003) tested a single pithos handle and found it effectively free of lipids. This makes sense for a storage vessel handle, especially if the

vessel was holding dried foods. It would not have been in contact with food covered hands in the same way, nor would food have boiled over or splashed onto it, as it would with a cooking or processing-type pot. Both Roumpou and colleagues' (2003) study and the current study suffer from extremely small sample sizes. A study of handles would be useful to test the findings from this and Roumpou and colleagues' study to compare known cooking pot handles to storage container handles.

Sauceboat Function

Sauceboats are important vessels of EBA II as both diagnostic and widespread in the period (Broodbank 2000:305; Caskey 1960; Fahy 1964; Pullen 1995). They are some of the most exquisitely made fine wares of EBA II and have been recovered from feasting, storage, and burial contexts (Fahy 1964; Broodbank 2000; Wiencke 1989, 2000; and many others) (Figure 5.2). They are thought to be connected to ritualized drinking practices (Broodbank 2000:306; Fahy 1964; Wiencke 1989). It has been argued that they must have garnered a specialized function, since pouring and drinking receptacles already existed in this period (Wiencke 1989:503). Sauceboats may have been "associated with a new product----fermented milk or wine" (Wiencke 1989:503). At Lerna, sauceboats were recovered in pairs and at some other sites, double-spouted examples have been found (Weinberg 1969; Wiencke 1989:505). This has led some researchers to reason that they were used for drinking between a pair of individuals (Weinberg 1969).

There is much debate about how the sauceboat functioned. It has been argued that one would drink directly from the spout (Weinberg 1969). However, Pullen (2011:224) has pointed out how awkward drinking from the spout actually is in practice, given its

sharp angle off the body. Theodorou-Mavromatidi (2007:248, 250) argued instead that the sauceboat had a double function as ladles for decanting liquids from larger vessels and as jars to pour liquids into smaller vessels. The spout was both used for pouring and as a handle. The actual handle, which is quite flimsy, would have functioned as a hook on which to hang the sauceboat between uses (Theodorou-Mavromatidi 2007). The lip on the spout could have allowed the sauceboat to hang on the inner lip of a larger vessel (Theodorou-Mavromatidi 2007:251).

I hypothesized that sauceboats were used for serving liquids and were connected to ritualized drinking practices by sampling from two sauceboats. I expected to find biomarker evidence for beverages, possibly alcoholic. Theodorou-Mavromatidi (2007) called for residue analysis to shed light on sauceboats over a decade ago. She mentioned that chemical residue analysis of sauceboats was being conducted at Akrotiri, but this research apparently remains unpublished to date. To my knowledge, these are the first sauceboats ever analyzed with organic residue analysis.

One sherd (2062) is certain to have belonged to a sauceboat, but its contents are surprising. This yellow-mottled sauceboat had been reconstructed. I sampled a piece that did not directly mend but was clearly a part of the same imported vessel. As mentioned in the last chapter, this sauceboat held a mixture of pine resin, more than one plant, and animal products. Two characteristics of this lipid profile are surprising if this vessel contained alcoholic beverages: the abundant cholesterol and the high level of unsaturation. The plant sterols total 3.7 μg/g and the cholesterol or its derivatives total 2.8 μg/g. It is hard to reconcile why cholesterol would be present in any beverage, alcoholic

Figure 5.2 Yellow-mottled sauceboat

or non-alcoholic. The cholesterol is not likely contamination, since there are multiple forms of cholesterol oxidative products found in this sample, none of which were found in the TLE or N blanks.

There are several possibilities that could explain this residue. Wiencke's suggestion of fermented milk would account for the presence of cholesterol, although the plant and resin component would then become curious. Also, milk should contain odd-chain fatty acids between C15:0 and C19:0 with branched versions of these fatty acids, while fermentation yields abundant microbial biomarkers (Gunstone and Harwood 2007:93; Isaksson et al. 2010). Sample 2062 contained $C_{15:0}$, $C_{15:0}$ branched, $C_{17:0}$, and $C_{17:0}$ branched. Possibly the vessel was used to drink multiple beverages at various times—a fermented milk beverage in some settings and a plant-based drink in another setting with pine resin as a flavoring in either. Stable isotope analysis would be necessary to confirm any residue with dairy. Another possibility would be that cholesterol and pine resin were added as post-firing treatment. Given how thin the sauceboat is, one would imagine that it would need to be sealed prior to successfully holding any liquid. In this scenario, the rest of the lipids could be all accounted for by a vegetable/seed oil. In fact, the large amount of unsaturated FA, plant sterols, and $C_{8:0}$-$C_{10:0}$ provide evidence for a vegetable oil.

A second possible sauceboat (5486/7370) was tested, and its contents were surprising for a different reason. Although 5486 and 7370 did not directly mend, it is believed that they both belonged to an Urfirnis-slipped sauceboat (Fanis Mavridis, personal communication 2018; Žarko Tankosić, personal communication 2018). This sauceboat might be an import, since Urfirnis sauceboats are thought to originate from the

mainland (Broodbank 2008:64). Petrographic analysis would be the only way to positively confirm this, since the fabric looks identical to the local fabric with the naked eye. As reported in the Results chapter, it had excellent preservation of a unique set of sterols and a limited range of other compounds. The limited alkanes and alkanols suggest only one plant resource. This particular suite of major and minor sterols is found in oils, particularly sesame, linseed, poppy, and wheat germ. None of the possible wine biomarkers are present and these unusual sterols are not found in wine.

The hypothesis that sauceboats are pouring or ladling vessels might be supported by the residue results of three other sherds that are not from sauceboats. Samples 2991, 12461, and 5112A were identical in their sterol content to each other and to 5486. Sample 2991 is a thick, flared rim with an Urfirnis slip. The sample was taken at the place where the rim is attached to the body. Sample 12461 is a pedestal base of similar thickness. Dark splotches may be the remnants of Urfirnis slip, but it is difficult to tell. Sample 5112A is a grey body sherd with a handle attached to the exterior. Its fabric is different from the three grey fabric sherds discussed previously, but it could possibly be an import as well. The vessel types that these sherds belong to are unknown. It is possible that there is a bulk container and serving relationship between these sherds and the sauceboat. The only difference between the four sherds is that the sauceboat and the flared rim sherd had pine resin biomarkers. Sample 2991 may have the best evidence for oil of the four, as it has a relatively high unsaturation level and the unique sterol content found in linseed, sesame, poppy, or wheat germ oil. These non-sauceboat samples were all found in Layer 4, which is the dense layer with macrobotanicals.

The unusually high sterol content found in these four sherds could be the product of a unusually high-sterol plant. Some of the oils that have been proposed have a higher sterol content than others. Sesame oil has 398 mg/g, linseed has 756 mg/g, and wheat germ oil 1543 mg/g of sterols (Kornfeldt and Croon 1981:308). For perspective, olive oil has 510 mg/g of sterols (Kornfeldt and Croon 1981:308). Linseed or wheat germ oil might be the best candidates from this list, as the higher sterol content in fresh oil might have fostered preservation archaeologically. Both linseed and wheat were known to be used in this period according to the palaeoethnobotanical record from the broader region. The earliest palaeoethnobotanical evidence for sesame seeds does not appear until the Late Bronze Age at Akrotiri (Sarpaki 1987). Poppy use is not attested in Greece until the Protogeometric period (Kroll 1983, 1993). As a side note, modern oil contamination on these four sherds is a possibility worth discussing, given how well the sterols are preserved. However, if modern oil did contaminate these sherds, we would not only see well-preserved and abundant unsaturated TAGs, but also much higher free unsaturated FA. No TAGs were found in any of these.

If we look at the two sauceboats together, it is plausible that they both contained vegetable oil, but with differential preservation. For some reason, the sterols could have preserved better in 5486 and the fatty acids preserved better in 2062. Sample 5486 is from Unit 2 in Trench 9, which was a pit of animal bones and pottery; its pair 7370 was found in unit 1 in the same trench, which was a concentration of human bone (Mavridis and Tankosić 2016a:233). The main differences between the contents of the two is that 2062 has abundant cholesterol and more than one plant represented, while only 5486 contained pine resin. A cholesterol-containing substance, such as animal lard, might have

been used as a sealant for 2062, which did not have interior slip like 5486/7370 had. The Urfirnis slip of the latter may have helped to seal the vessel, so that it would not need additional products applied. The plant residue deriving from a sole source in 5486/7370 accords well with a single oil as its contents. Admittedly, the evidence for more than one plant in 2062 is difficult to explain and the presence of cholesterol has multiple equally valid explanations. Alternatively, if one sauceboat was used to serve an edible oil and the other mixture of substances or multiple beverages at different times, these vessels are no longer homogenous entities.

The connection of these vessels exclusively to drinking practices is called into question if at least one of them held a plant oil. Maybe this oil was a scarce and prized commodity that warranted a specialized vessel to serve as conspicuous consumption. This oil could still be linked to feasting, in that it was maybe poured over foods as a condiment. Gold of pleasure oil, for example, is often used as a flavoring (Megaloudi 2006:59). Why these would be found in pairs remains a question. Maybe everyone had a small amount of this oil and vessel of their own they would bring for themselves or their household. In fact, we have found at least four sauceboats at Ayia Triada that could represent four individuals or households participating. Or we could envision these vessels and oils not for consumption at all. Oil, in particular olive oil, was used by the Classical Greeks to cleanse the body. It is equally possible that the seed oil likely contained in the sauceboats was used as a cleansing or purification ritual for the body. Then, the spout would be appendage for pouring, not to directly drink from. The linkage of sauceboats with burial contexts would lend support to this suggestion as would the presence of bone

tubes, which were found at Ayia Triada. These ornamented, pen-like objects were possibly used for pigment application or bodily rituals (Mavridis and Tankosić 2016a).

Until more sauceboats are tested to see which if any of these patterns persist, we cannot make broad assumptions yet about these vessels.

Summary

To conclude this discussion, I highlight the results from my proposed hypotheses. Regarding the first hypothesis, there is no evidence to suggest that the figs, peas, lentils, or grains recovered as possible feasting remains were used in the serving vessels, although the lack of robust biomarkers hinders their identifications. The short and squat storage jars likely held different contents besides these plant foods, since they all had substantial amounts of lipids. The second hypothesis regarding the supposed liquid storage has at least some evidence to support it. We have no evidence that the liquids were specifically vegetable oils or alcoholic beverages. However, the ovoid type's overall low lipid content and its lipid distribution possibly suggests a lipid-poor liquid. The drainage hole and presence of beeswax in the bulbous type could support that it held liquids, if beeswax was used as a sealant. The third hypothesis is refuted, as variability was certainly found within the bowls and jars. The bowls varied in the range of plant resources represented, while one bowl had only plant residue. The storage jars had different commodities as well, mostly beeswax and pine resin found only in some of the examples. My hypothesis that imported wares contained different contents than local wares cannot be substantiated. Some of them may have had smaller quantities, but so did some local wares. This may be more a reflection of these sherds being fine wares than

them being imported wares. In terms of the handle hypothesis, I have proven that handles cannot always be used as controls for soil contamination. The function of some vessels could predispose them to lipid absorption. Finally, for the hypothesis concerning the sauceboats, at least one sauceboat could have been linked to the drinking of beverages, possibly a fermented milk or plant-based drink. However, it is also possible that both sauceboats held seed oils that were meant to be consumed or were related to ritual cleansing.

CHAPTER SIX
CONCLUSION

I employed a systematic residue sampling strategy at Ayia Triada Cave to identify the original contents and resource use patterns within the pottery, in an effort to expound upon feasting in burial contexts. I outlined several scenarios in the Introduction that could have emerged from the residue data. The vessels could have been created specifically for burial with the dead and deposited empty. Secondly, they could have held a narrow range of foodstuffs reserved exclusively for burial feasting and therefore reflect specialized consumption practices. Thirdly, the pottery assemblage could contain a wide range of food items that were served or stored in intensively used vessels. The second and third scenarios could shed light on foodways of the community to which the participants belong, while all three scenarios could illuminate attitudes and behaviors toward death and larger cultural values.

The picture that has emerged from the residue analysis conducted at Ayia Triada is a complex one. The data suggest that these were not pristine, never-used vessels; instead the third scenario of frequently used vessels seems most plausible. Bowls contained a range of substances, with some indicating multiple plant resources and nearly all with an animal component. The storage jar residues reflect a range of foods and/or liquids stored within them as well. Although the exact identity of the contents cannot be determined, the presence of beeswax and/or pine resin in certain storage jars points to specialized storage. These jars either held beeswax, honey, or a commodity that required beeswax as a sealant. Pine resin was possibly an ingredient in the jars in which it was

found. The one possible cooking vessel contained a mixture of plant epicuticular wax, plant resin, and animal resources. Several vessels, including one sauceboat, have strong sterol evidence for a vegetable oil, possibly linseed oil. The yellow-mottled sauceboat could have held oil and/or have been used for multiple beverages, one of which might have been served during the feast. Footed cups were excavated from the same EBA II layers, but were not sampled; they could have been the individual receptacles for the yellow-mottled sauceboat's contents. Most of these vessels, besides the sauceboats and a few imported wares, appear to be vessels that would have been a part of any domestic assemblage at the time.

We see a continuation of these patterns in the unknown vessel sherds. The majority of these sherds contained mixtures of plant and animal resources. They too appear to be heavily used. In fact, the samples with the three largest residue quantities were from unknown vessel types. Like most of the sherds from known vessels, the chemical signatures appear to either represent mixtures of various foodstuffs or repeated use of the vessels with different substances. A few fine wares and imports appeared to be less utilized, but with so few sampled in this study, it is difficult to extrapolate a trend.

The residue data suggest that these vessels were not made exclusively for burial. Rather, they appear to have been used in everyday life, because of the breadth of use displayed in their residues. Only four sherds lack quantifiable residues, but even these could be explained: one sample came from position on the vessel not likely to have residue; a thick slip prevented lipid absorption in another; and the other two could indicate low lipid or dried contents were held within. Although it is possible that the vessels deposited in the cave were used for the one and only time during the burial ritual,

one episode would probably not have produced abundant lipid signatures. The patterns of most samples at Ayia Triada were likely created with heavy, regular, and probably additive use. It is estimated that only 1% of the original lipid survives through archaeological time (Evershed 2008b:28). Such high recovery rates among these samples (94%) and their generally abundant residues suggest that there would have been considerable residues to begin with if what remains represents only a minute amount of what was once there. This is especially true for the compounds of plant origin, for which we have many strong signatures. Given that plants generally contain lower lipid quantities to begin with, multiple episodes would be needed to produce appreciable and abundant plant residues. The heavy use, the range of foods exhibited, and the variability within vessels, especially the bowls and storage jars, evoke everyday domestic context, rather than use of specialized pots or foodstuffs.

These results generally concur with the argument that feasting occurred in the cave, which was suggested by Mavridis and Tankosić (2016a). The participants of the funerary rituals consumed a wide range of foodstuffs, including edible oils. We have a cooking jar and individual serving vessels. The bowls may have served some of the foods physically preserved in the cave, as the animal component of the residues could relate to the sheep/goat bones found. The oil that is identified in the sauceboats and few other sherds could have been poured over the participants' food for flavoring. The presence of widespread yeast breakdown products might support a meal being consumed in the cave and left. The leftover foods were likely not washed out of the vessels and were deposited 'dirty', in which case the natural yeast on the foods themselves or in the cave could have immediately begun to act on the residues. The feasting participants might have cooked in

the cave, since one vessel has evidence for cooking and there is evidence for burning/fire. However, given how dark and cramped this part of the cave is and how quickly the air would become too thick with smoke to breathe, it seems unlikely. It seems reasonable that the meal had been cooked elsewhere, the full cooking pot (or pots if more are identified in the last set of samples) and storage jars were transported, and then the meal was consumed prior to the burial of the deceased. The foods were then dumped, ritually burned, and the human remains were laid out on top. The ritual killing of at least the storage vessels, which were smashed *in situ*, ensued and the sherds were deposited at the edge of this activity (Mavridis and Tankosić 2016a:234). The participants then left the cave, possibly to return later to partially collect the bones for burial in a final location, unfortunately one that remains unknown.

Feasting signatures in the archaeological record can reveal the form and function of the feast (Hayden 2001). A small feast is usually indicated archaeologically only by a special location that is not domestic, much like Ayia Triada Cave (Hayden 2001:40, 46). The number of small bowls/saucers (likely the individual serving unit) I observed in the assemblage and many of which I sampled agrees with this assessment of feasting size, although one caveat should be made. My sample is only a portion of the original assemblage and the final serving ware counts are not yet available. This feasting event likely represents a solidarity feast within a group, indicated by "minimal departures from standard daily foods or material items" (Hayden 2001:38). Hayden (2001:39) suggested that the larger number of feast participants, the more tailored the foods and vessels become. This is the opposite of what the residue data indicates here. We do not see evidence for highly specialized foods, besides possibly the vegetable oil, or a large

number of special vessels. There is nothing to suggest different communities were involved here. We can envision feast of everyday foods was consumed at the time of the burial. A conscious linking of the feast with the everyday could be powerful and transformative in this situation.

The depositional data is seemingly in conflict, therefore, with the residue data. The intentional manner in which human bones, grave goods, pottery, and organic remains were deposited in a special, non-domestic location point to ritualized behaviors, as the excavators noted (Mavridis and Tankosić 2016a). However, in this cave, we have a major component of the ritual, the food and heavily used pottery, that looks remarkably ordinary. This forces us to reevaluate our cultural and personal biases of what ritual is supposed to look like. We often assume that every element of a ritual must be a departure from the norm or the non-ritual. It needs to be special or different to hold any importance or symbolism, but clearly this is not always the case.

These findings make us question why individuals would transport the deceased, copious amounts of foods, and used, possibly full vessels from their home into a mountain cave, consume a meal in a deep and dark area of this cave, and then bury the meal equipment and leftover foods with corpses resting on top. Residue analysis certainly cannot reveal the motivations of individuals, but we can see deliberate choices they made and extrapolate *habitus* from these choices. The practice of producing, processing, and consuming meals daily created habits, which were then reified in the cultural consciousness. These everyday practices embody social relationships. In an effort to reinforce the cultural order of the family or community, these people might have recalled the everyday foods and vessels for this burial feast, but then elaborated on them with the

fine-ware sauceboats and other fine-ware vessels. The special vessels might have been used for status display. The sauceboats may have held valuable edible oils, beverages, or been used for the ritual cleansing or purification of the bodies of the living or the deceased. Modern day American funerals are not much different from this feasting event other than location, where food also is served in relation to a burial, but the recipes are basically prescribed from the everyday in the cultural or subcultural mindset.

This association with the everyday does not negate that it may have held meaning for the participants nor does it prevent us from drawing initial conclusions about the community to which the participants belong. EBA II was a time of greater social complexity, although scholars do not fully agree on the societal organization. Differing patterns have emerged from Mainland Greece and the Cycladic islands. The mainland was possibly a series of chiefdoms, while most of the Cyclades were likely more egalitarian (Broodbank 2008; Pullen 2008; Wiencke 1989). There was certainly interaction between these two worlds, as an increase in maritime trade was evident in EBA II (Broodbank 2008; C. Renfrew 1972; Kouka 2008). Where exactly Southern Euboea fits into this picture is unclear. Settlements are scattered across the landscape and are not densely distributed. Most sites are located on the coast, are seasonal, and short term, although there are three larger, more permanent settlements (Cullen et al. 2011,2013). The material culture displays a blending of Helladic and Cycladic characteristics, which makes sense given its locus at maritime crossroads. Southern Euboea functions almost like an island, because its geographical position and topography (Mavridis and Tankosić 2016a; Tankosić 2017).

The linking of daily food practices to an important rite of passage ritual like funerary feasting suggests that collective identity was important to the group to which the participants belong. The term group is meant to be fluid term here, as Mavridis and Tankosić (2016a) have also suggested; it could mean the community, a lineage, or merely a nuclear family. We still cannot identify the settlement to which these people belong, and so cannot clarify the composition of the group represented here. Regardless, their identity is embedded in these daily food practices and it seems they are intentionally signaling it in the funerary rituals. In this fractured cultural and geographic landscape, people may have felt an even greater need to stake a claim their own identity and to assert collective membership. By incorporating domestic foodways, which are reinforced daily, it also suggests that they place value on the inherent social relationships that are embedded. Barrett (1991:6) underscores this interpretation: "ritual and religious knowledges thus are built out of the same material conditions as everyday life; they cannot be analyzed as though they somehow have a life of their own."

Limitations

There are a few limitations that should be noted. One such limitation is in the data itself. Potential data was lost, since a fatty acid fraction of the results was unusable in all samples. Biomarkers could have been extracted in the fatty acid component, such as those that indicate the processing of fish resources, isoprenoid fatty acids and ω-(o-alkylphenyl)alkanoic acids (Baeten et al. 2013; Hansel et al. 2004). Additionally, the $C_{18:0}/C_{16:0}$ ratio can broadly distinguish between plant and animal resources. Even though fatty acids were detected in the TLE, ratios can only be calculated from the fatty acid

fraction. This ratio would have been most useful in characterizing the less abundant residues or those without sterols, although the number of samples that fell in this category is relatively small.

Another limitation is methodological concerning the identification of animal resources. It is not possible to distinguish fatty substances originating from ruminant adipose fat, non-ruminant adipose fat, or dairy with GC/MS alone (Evershed et al. 2002). Having this information would be valuable for many sherds, especially for one of the sauceboats that possibly contained dairy products. The other main limitations are merely circumstantial. Since the pottery analysis is still in progress, I cannot yet analyze these sherds/vessels relative to entire pottery assemblage nor can I identify types to which some sherds belong. Additionally, since the macrobotanical remains and animal bones have not been fully analyzed, I can only partially compare the residues to the physical remains.

Future Research

Future research can expand the findings of this study. The remaining 42 samples once analyzed can hopefully speak to the general trends discovered thus far in the residue data. These samples were excavated from the same EBA II levels, and therefore I expect to observe the same intensity of use and variation of compounds. Several sherds that await analysis form a pair with sherds that have already been analyzed. The analysis of these pairs can further reveal the spatial distribution of lipids across their respective vessels and determine vessel function.

Several exciting avenues for future research have arisen as a result of the findings so far. The presence of labdane remains a mystery. Analyzing samples from other time periods and areas in the cave could determine if labdane originated from within the site and if it was exclusively linked to the EBA II activity, which could possibly suggest another phase of the ritual. Alternatively, analyzing pottery from other sites that was processed simultaneously at the same facility as Ayia Triada's pottery could determine if post-excavation processing was the culprit. Now that I have a data set for residues in a non-domestic, likely ritual context, I would like to compare residues from domestic contexts in Southern Euboea to determine if the consumption patterns are indeed similar. Since it is confirmed that animal fats are preserved, it would be now beneficial to conduct stable carbon isotope analysis of Ayia Triada samples. This could reveal the presence of dairy and any other animal species that were not present in the zooarchaeological remains. A particularly promising area of future research is with the sauceboats. The surprising results justify future analyses of the other sauceboats at Ayia Triada and those at other EBA II sites as well. This study confirmed for the first time that lipids do survive in these vessels, but the findings suggest several possibilities with respect to their contents and possible function. It would be beneficial to search for vegetable oil, animal products as sealants, fermented dairy beverages, plant resin, and/or combined substance use within these vessels.

As already mentioned, several analyses are still ongoing within the Ayia Triada Cave project, particularly macrobotanical, faunal, and ceramic analyses. Once completed, these analyses can lend more support to my conclusions. Once the ceramic analysis is conducted, I would like to compare my results with the use wear patterns of the pottery.

This could provide supplementary evidence for the heavy use of vessels. With the ceramic analysis completed, I will be able to further characterize the unknown vessel sherds by type. It will be interesting to see if the variability within vessel types continues. Also, the ratio of types within serving ware, namely the number of individual bowls to larger serving bowls, can shed light on the number of people participating in the feast (Hayden 2001:47). Combining this information with the residue data may allow for personal serving sets to be identified. Lastly, complete floral and faunal species lists will provide a broader picture of plant and animal exploitation and identify other species for which I can search in the remaining samples.

The last area of future research is in relation to a relatively new finding. Possible cereal biomarkers have been recently published, over a year after I completed the extraction and GC/MS work for this study (Colonese et al. 2017; Hammann and Cramp 2018). It would be valuable to adopt a targeted approach to identify cereals within Ayia Triada pottery. I would collect additional samples from the cooking pot, the larger bowl, the small bowls, and a selection of storage jars and subject them to the enrichment protocol and ultra-sensitive time-of-flight GC/MS to search for these biomarkers (Hammann and Cramp 2018).

Summary

The analysis of organic residues from pottery has allowed us to glimpse ritual behaviors surrounding the burials at Ayia Triada Cave. However, as is so often the case in archaeological research, this analysis elicits more questions than answers. The fact that these residues were so complex and that it is often impossible to tease out individual

resource signatures speaks to the complexity of human behavior. We are seeing complex ritual behaviors surrounding the burials, which are themselves layered with meaning, social interactions, collective consciousness, and personal experiences. However, we are also likely witnessing months and maybe even years of practices and behaviors surrounding foodways that are recorded in these residues. The pots may or may not have served or stored the physical food remains left in the cave, but we do know that the vessels were certainly plucked from everyday life. “Food has the constant tendency to transform itself into situation” (Barthes 1997:26) and this is precisely what I envision happened in Ayia Triada Cave when nine individuals were either simultaneously or intermittently buried.

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APPENDIX

Sample	Quantity (µg/g)	Pottery Type	Trench	Level	Unit	Residue description	Interpretation
129	76.2	Fine ware, globular pyxis with buff fabric; possibly painted; base sherd	9	4		Cholesterol, cholest-5-en-7-one, and β-sitosterol; more MAGs than FA, although very small range of both; small amount of $C_{16:1}$ and $C_{18:1}$ FA and abundant MAG 15:1; abundant branched alkanols and alkanes; alkanes range from 19-33 and peak at 23-24; not as wide of range of the plant alkanols (OL 20, 22, 24), but wide range of <20 OL; OL 18 is the most abundant alkanol; some secondary alcohols	Mixture of highly degraded plant wax, plant/fish, and animal resources. Small range of fatty acids and MAGs suggest limited sources.
653	88.0	Large, coarse ware footed base	8	4	1	Small range of FA ($C_{12:0}$-$C_{18:0}$ even and $C_{13:0}$); no unsaturated FA; saturated MAGs and DAGs; MAGs fairly abundant; some branched alkanes and alkanols; small range of alkanols from OL12-16, 18, 22; OL 16 and 18 are the most abundant alkanols; alkanes peak at alkane 20; no sterols; neutral fraction unusable	Indeterminate, degraded residue.
1656	133.6	Medium coarse handle	4	4		Beeswax components in the TLE: $C_{24:0}$, odd dominated alkanes 31>29>27, odd alkenes, abundant OL 24-34 even, OL 30 and 32 have the greatest quantity of all compounds in the TLE, $C_{16:0}$ WE 40-48, secondary alcohols amongst WE, α-(ω-1) diols; cholesterol and 7-ketocholesterol; 15-hydroxy-7-oxo-DHA; some saturated MAGs; small quantity of DAGs; range of FA from $C_{8:0}$-$C_{25:0}$ (missing $C_{13:0}$, $C_{19:0}$, $C_{21:0}$); abundant $C_{14:0}$ and $C_{18:1}$; lesser quantities of $C_{16:1}$ and $C_{9:0}$, which indicates breakdown of $C_{18:1}$; $C_{15:0}$ and $C_{17:0}$; numerous branched alkanols indicate microbial degradation; WE 20:0 and 16:1; unsaturated 1-tetracontanol, 1-dotriacontanol, and 1-eicosanol; glycerol present in TLE	Complex mixture of beeswax, pine resin, plant or fish ($C_{16:1}$) and animal resources with microbial degradation. Large range of fatty acids suggests mixture of fatty sources. Good preservation.

1686	66.5	Coarse, yellow-mottled jar, body sherd	4	4		Mostly alkanols and FA, including $C_{15:0}$, $C_{18:2}$, $C_{18:1}$, and $C_{16:1}$; unsaturated FA total 7% of the residue; limited range of saturated FA; only alkanes are 18, 28, 32; some saturated MAGs; cholesterol and Δ^5-avenasterol; wide range of even alkanols from OL 22-32, although not abundant; alkanol sequence also includes 12-16 and 18; OL 18 and 16 are the most abundant alkanols; neutral fraction unusable.	Mixture of plant wax, animal, and plant or fish ($C_{16:1}$ and $C_{18:2}$) with some microbial degradation; wide range of alkanols with no clear principle homologue(s) suggests multiple plant sources
2062	204.5	Fine ware, yellow mottled sauceboat, body sherd	8	4		Stigmasterol, β-sitosterol, Δ^5avenasterol, cholesterol, 7-ketocholesterol, cholesta-3,5-dien-7-one; 15- hydroxyl-7-oxo-DHA; FA C8:0-18:0 and 20:0; branched FA C13:0, 15:0, 16:0, and 17:0; multiple branched C15:0 and C17:0; abundant C18:2, 18:1, and 16:1 in two isomers (6% of TLE); saturated MAGs and DAGs relatively abundant; diacids C6-7 (possibly); alkane 18-36 present, peaking at 24; wide range of alkanols OL 22-32; OL 13-20 also present; most abundant is OL18; several secondary alcohols; branched OLs	Mixture of pine resin, plant epicuticular wax, and animal products with microbial degradation. More than one plant resource. $C_{18:2}$ and $C_{16:1}$ could originate from plants or fish.
2991	57.3	Medium coarse pedestal base	8	4		Diverse and abundant sterols with β-sitosterol dominating; Δ^5avenasterol, unidentified sterol, poriferasta-7,25-dienol, cyclolaudenol or 24-methylene cycloartenol; stigmasterol; unidentified stigmasterol relative; campesterol; cycloeucalenol, cycloartenol, or oleana-11,13(18)-diene; small amount of cholesterol Retene and 9,10-Anthracenedione Moderate range of FA $C_{8:0}$, $C_{9:0}$, $C_{12:0}$-$C_{18:0}$, $C_{20:0}$; unsaturated FAs ($C_{18:1}$, $C_{16:1}$, $C_{18:2}$) make up 1/3 of total fatty acids; $C_{15:0}$ and $C_{17:0}$ suggest microbial	Unique plant residue with diverse sterols and pine pitch. Microbial degradation of the fatty substances. Relatively large amount of unsaturated FA. $C_{18:1}$ could originate from plant or animal. $C_{16:1}$ and $C_{18:2}$ could originate from plant or fish. Possibly a vegetable oil.

						degradation; some saturated MAGs and trace saturated DAGs; alkanes range from 18-27, 29, 31; alkane 22 is the most abundant; OL 20-26 in low amounts; OL 12-16, 18-19 also present; OL 18 most abundant; two secondary alcohols; minor branching of alkanes and alkanols	
3035	62.6	Coarse base/body with sooting on the interior	8	4		K31 and K33; cholesterol; β-sitosterol; ketone 2-heptadecanone; wide range of even and odd FA from $C_{8:0}$-$C_{32:0}$, including $C_{15:0}$, $C_{17:0}$, $C_{19:0}$; small quantity of two $C_{18:1}$ isomers; decent amount of mostly saturated MAGs and some DAGs; n-heptadecan-1,2-diol; WE with $C_{10:0}$, $C_{14:0}$, and $C_{16:0}$ FA components; full range of alkanes 18-33, peaking at alkane 23; odd alkenes; even OL 22-34, but no major homologue(s); OL12-19 present; OL 18 is most abundant OL; minor branched alkanes and OLs; diacid C6	Mixture of plant epicuticular wax and animal sources that was heated and microbially degraded. Fatty material originating from multiple sources. Multiple plant sources represented. Good preservation indicated by $C_{8:0}$-$C_{10:0}$ and adipic acid.
3038	207.1	Medium coarse, short and squat jar, body sherd	8	5b		WE 40-46 even, even-dominated OLs 24, 26, 28, 30, and 32 (OL26 is the most abundant), odd-favored alkanes with 31 being the highest, $C_{24:0}$, abundant $C_{16:0}$, one α-(ω-1) diol; β-sitosterol and germanicol; abundant cholesterol; 15-hydroxy-7-oxo-DHA; abundant saturated MAGs/DAGs; abundant saturated FA $C_{12:0}$-$C_{24:0}$ even and $C_{15:0}$, $C_{17:0}$, $C_{19:0}$; branched $C_{15:0}$; $C_{18:1}$ in two isomeric forms and $C_{16:1}$ totaling 3% of TLE; OL 12-19 also present; fair amount of OL 18; many branched OLs and some branched alkanes; some secondary alcohols; unsaturated OL 20:1	Degraded mixture of pine resin, beeswax, plant, and animal. $C_{16:1}$ could indicate plant or fish.

3040	114.3	Medium coarse bowl, body sherd	8	5b		Cholesterol and β-sitosterol; small range of even FA $C_{14:0}$-$C_{18:0}$, and $C_{22:0}$; small amount of $C_{18:1}$; MAG 16:0 only; alkanes 18-33, peaking at 24; many branched alkanes; range of alkanols; OLs 20, 22, 24; OL 12-19 also present; OL 18 is the most abundant; some branched OLs; many secondary alcohols; unsaturated OL 20:1 and 22:1; abundance of alkanes and OLs	Mixture of plant and animal with microbial degradation.
3049	32.7	Coarse body sherd	8	4		DHA and 7-oxo-DHA, methyl ester; β-sitosterol, stigmastanol, cholesterol, and possibly ergosta-7,22-dien-3β-ol; stigmasta-3,5-dien-7-one; relatively large amount of secondary alcohols (5% of N); OL 13-28 (except 17) with some branching; even-dominated OL20-28; OL 18 is the most abundant; many branched alkanes; extensive range of odd-dominated alkanes with alkane 31 dominating; one WE; small amount of saturated DAGs/MAGS and FA $C_{6:0}$-$C_{18:0}$ saturated; $C_{18:1}$	Mixture of degraded pine resin, meat, and plants with microbial degradation. Range of alkanes and alkanols suggests multiple plant sources. Good preservation, because of the small FA present.
3133 A	1001.4	Coarse body sherd	8	4		Very abundant alkanes 27>29>31>33; abundant $C_{24:0}$ and $C_{16:0}$, WE 40-46, secondary alcohols amongst WE, very abundant even OL 24-36, especially OL 30; abundant α-(ω-1) diols and two α-ω diols Small quantity of MAGS/DAGs; FA range from $C_{8:0}$-$C_{24:0}$, but are not particularly abundant; some $C_{18:1}$; DHA; decanal; 2-decanone, 2-undecanone, and 2-dodecanone; oxalic acid OL 12-19 also present in low abundance; WE 20:0 dicarboxylic acids C6,9,10 with 9 the most prevalent; no sterols	Beeswax with *Rue*, pine resin, and unknown fatty substances that have been degraded, possibly vegetable oils. Degradation is evidenced by odd FA and branched alkanes and OLs.

3198	38.7	Medium course shoulder	8	4		Trace cholesterol (0.01 μg/g); small quantity of saturated MAGs; small range of FA ($C_{12:0}$, $C_{14:0}$-$C_{18:0}$ only); many branched alkanes; full alkane range from 18-32; somewhat odd-dominated from 26-32, but alkane 24 is the most abundant; only even OLs from 20-32; OL 12-19 present; OLs peak at 18; some branched OLs	Possibly animal residue with plant epicuticular wax from multiple plants.
3275	18.2	Medium coarse body	8	4		Small quantity of cholesterol; small range and low abundance of saturated FA $C_{12:0}$-$C_{18:0}$ even; small amount of saturated MAGs; OL range is not extensive: OL 12-16, 18-20, 22 with some branched; OL 18 is the most abundant; many branched alkanes; even dominated alkanes peaking at 24; alkenes 21:1 and 23:1 9,10-anthracenedione	Primarily meat and possibly degraded plant residue?
3292	164.7	Medium coarse, short and squat jar, body sherd	8	4		$C_{24:0}$; abundant $C_{16:0}$; OL 22-34 even, with OL24 the most abundant; 16:0 WE 42-46 even; odd-dominated alkanes from 23-33; some branched alkanes; alkane 19 and 20 are the most abundant; abundant saturated DAGs/MAGs; wide range of FA $C_{9:0}$-$C_{18:0}$ and $C_{20:0}$-$C_{28:0}$ even; two isomers of $C_{18:1}$; branched $C_{13:0}$, $C_{15:0}$, and $C_{17:0}$; numerous branched alkanols; OL13-14, 16-19 also present; OL 18 the most abundant; 15-hydroxy-7-oxo-DHA; cholesterol, β-sitosterol, and germanicol; other WE $C_{16:1}$ and $C_{14:0}$.	Complex mixture of plant, animal, and pine resin with significant microbial degradation.
3482	149.9	Coarse body of handled open vessel with flaring rim	12	4		K31, 33, 35; wide range of alkanes 18-36; alkane 20 is the most abundant; alkenes and some branched alkanes; wide range of alkanols with OL12-26, 28, 30, 32; OL18 highest; β-sitosterol, cholesterol, 7-ketocholesterol, and cholesta-3,5-dien-7-one; 15-hydroxy-7-oxo-DHA; wax esters of 14:0, 16:0, and 16:1 FA components; even and odd FA from $C_{8:0}$-$C_{26:0}$ (except $C_{13:0}$, $C_{20:0}$, $C_{22:0}$); $C_{16:1}$	Complex mixture of plant epicuticular wax, pine resin, plant/fish ($C_{16:1}$), and animal products that was heated. Bacterial degradation present. Multiple plants

						and $C_{18:1}$ in small amounts; straight chain and branched $C_{15:0}$ and $C_{17:0}$; saturated MAGs/DAGs in small abundance; K27	represented and wide range of fatty materials.
4469	118.6	Bulbous, medium coarse jar with hole, body sherd	9	5a		Odd dominated alkanes 31>23>29>27>25>33; $C_{16:0}$ WE 40-48; even FA $C_{22:0}$-$C_{28:0}$; $C_{24:0}$ the most abundant within that range; abundant $C_{16:0}$; OL 24-34 even; wide range of FA from $C_{9:0}$-$C_{18:0}$ (except $C_{10:0}$ and $C_{17:0}$) ; $C_{15:0}$ branched; $C_{16:1}$ and $C_{18:1}$; saturated MAGs/DAGs; 15-hydroxy-7-oxo-DHA; cholesterol; β-sitosterol; adipic acid; OL 12-16, 18-20 also present; OL 18 is the most abundant OL; wide range of alkanes from 18-33; branched OLs and alkanes	Possibly degraded beeswax mixed with pine resin, meat, and plants. Multiple plants and fatty substances are present. Some microbial degradation. $C_{16:1}$ could originate from plants or fish.
4972	111.3	Coarse, short and squat jar, body sherd	9	5a		Germanicol, cholesterol, and 7-ketocholesterol; stigmastane; DHA; wide range of FA $C_{6:0}$-$C_{26:0}$ (except $C_{7:0}$, $C_{21:0}$ and $C_{25:0}$); branched $C_{17:0}$ and $C_{15:0}$; $C_{18:1}$, $C_{16:1}$, and MAG 18:1; more $C_{18:1}$ inferred from presence of $C_{9:0}$; fairly abundant MAGs; low abundance of saturated DAGs; WE $C_{16:0}$ and $C_{16:1}$; wide range from alkane 18-33, peaking at alkane 22; alkenes 21:1 and 23:1; many branched alkanes; some branched OLs; wide range of OLs to OL30; OL16 and 18 most abundant; diacid C6	Mixture of pine resin, plant epicuticular wax, plant/fish ($C_{16:1}$), and animal products with microbial degradation. Wide range of OLs suggests more than one plant. Wide range of FA suggests multiple fatty sources.
5025	45.0	Medium coarse, short and squat jar, body sherd	9	5a		Cholesterol, stigmasterol, β-sitosterol, Δ^5avenasterol, and 7-ketocholesterol; 15-hydroxy-7-oxo-DHA; odd-dominated alkanes where 23>31>27>29>25>33; full range from 18-33; OL 22-28 in low abundance; OL 13-19 present; OL18 is the most abundant; saturated MAGs and small amount of DAGs, FA $C_{16:1}$ (two isomers), $C_{18:2}$, $C_{18:1}$ (two isomers); branched and straight chain $C_{15:0}$ and $C_{17:0}$; FA $C_{12:0}$-$C_{18:0}$ even; branched OLs and alkanes	Mixture of plant, animal, pine resin, and plant/fish ($C_{16:1}$ and $C_{18:2}$) with microbial degradation. Multiple plants represented.

5112 A	15.5	Medium coarse body sherd with handle attached	11	4		Most abundant compound in both the TLE and N is β-sitosterol; stigmasterol, Δ^5avenasterol, and campesterol; poriferasta-7,25-dienol, cyclolaudenol or 24-methylene cycloartenol; unidentified stigmasterol relative; cycloeucalenol, cycloartenol, or oleana-11,13(18)-diene; $C_{18:2}$, $C_{18:1}$, and $C_{16:1}$ comprise 10% of the TLE; tight range of FA otherwise with $C_{14:0}$, $C_{16:0}$, $C_{18:0}$; some saturated MAGS; wide range of alkanes 19-32, peaking at 23; some branched alkanes; OL 20-28 even in low abundance; OL 18 is the most abundant of all OLs; minor branched OL	Unique plant residue with diverse sterol profile and unsaturated FA originating from plant or fish. $C_{18:1}$ could originate from plant or animal. Possibly a vegetable oil.
5114	33.2	Coarse body sherd	11	4		Cholesterol, cholesta-3,5-dien-7-one, 7-ketocholesterol and β-sitosterol; 15-hydroxy-7-oxo-DHA; smaller range of FA from $C_{7:0}$-$C_{18:0}$ (except $C_{11:0}$); straight and branched $C_{15:0}$; some $C_{16:1}$ and $C_{18:1}$; MAGs and small amount of DAGs (all saturated); 16:0 and 14:0 WE; alkane 18 and 20-33, peaking at 23; numerous branched alkanes and OLs; some secondary alcohols; even dominated alkanols OL 20-32; OL 15-19 with OL18 as the most abundant of all alkanols; possibly K30; Manoyl oxide	Mixture of pine resin, plant epicuticular wax, plant/fish ($C_{16:1}$) and animal products with microbial degradation. More than one plant represented.
5175	458.7	Medium-large bowl with inturned rim; medium coarse, burnished, body sherd	11	5b		Dominated by alkanes and alkanols (58% total); alkanes peak at 24 and range from 18-33 (except 19); wide range from OL 22-34 even, dominated by OL30; OL 11-16, 18, 19, 21 are also present, with OL 14 as the most abundant; small range of saturated FA, dominated by $C_{14:0}$; $C_{9:0}$ indicates original presence of $C_{18:1}$; many branched alkanes and alkanols; some MAGs, including 16:1 and 18:1; some unsaturated alkanols (20, 22, 24) and alkenes; $C_{16:0}$ wax esters from C40-48 even; C16:1 WE also; o-coumaric acid; β-sitosterol*; largest compound is a secondary alcohol; large quantity of two	Complex, waxy mixture from multiple plant sources and animal products with moderate microbial degradation of wax components and mostly hydrolyzed unsaturated. Possibly plant resin?

						secondary alcohols; cholesterol and cholesta-3,5-dien-7-one, stigmasterol, and Δ^5 avenasterol; 10.26 μg/g of labdane; neutral is unusable	
5217	44.8	Coarse ware body sherd	9	5b		β-sitosterol*, stigmasterol, and Δ^5-avenasterol; only $C_{16:0}$ FA; saturated MAGs; alkane 31 is the only abundant alkane; wide range of alkanes from 18, 20-27, 29, 31; some branched alkanes; small range of alkanols from OL 12-20, except 17 and 19; OL 16 and 18 are the most abundant alkanols; Neutral is unusable	Plant residue, likely representing only one or two plants, with microbial degradation.
5278	13.4	Medium coarse rim sherd	9	5a		Cholesterol and β-sitosterol; minimal range of FA ($C_{14:0}$, $C_{16:0}$, $C_{18:0}$ only); saturated MAGs; small amounts of OL 22-30 even; OL 12-19 also present; OL 18 the highest; some branched alkanes and alkanols; alkanes 20-27, 29, 31, 33, peaking at 22	Small amount of plant and meat residue with slight microbial degradation. More than one plant likely.
5436	9.0	Coarse, ovoid jar, yellow-mottled, body sherd	9	4		Cholesterol,7-ketocholesterol, β-sitosterol, Δ^5 avenasterol; odd chain alkanes dominated by alkane 31; small amount of saturated DAGs/MAGs; small amount and range of FA $C_{9:0}$, $C_{12:0}$-$C_{18:0}$ even; $C_{15:0}$ and $C_{17:0}$; $C_{18:1}$, $C_{18:2}$, and $C_{16:1}$; several alkenes and branched OLs; even dominated OL 20-28; unsaturated OL 20; OL12-17 and OL 18 also present; OL18 has highest quantity	Plant wax and animal residue with microbial degradation. Wide range of plants represented by OLs. C18:2 and 16:1 originate from plants or fish.
5448	6.2	Coarse jar, body sherd	9	4		Mostly FA in a small range of $C_{12:0}$-$C_{18:0}$; $C_{15:0}$ and $C_{17:0}$; trace cholesterol; small amount of saturated MAGs; very few branched OLs and alkanes; alkenes 21:1 and 23:1; alkanes peak at 20, but not abundant; OL 20, 22 in low amounts; OL 12-16, 18 also present; OL 18 is the most abundant	Very small amount of residue, possibly animal residue and one-two plants?
5475	171.1	Medium coarse base	9		2	Abundant even dominated OLs from 24-36, making up 70% of TLE; $C_{22:0}$ and $C_{24:0}$; abundant $C_{16:0}$; odd dominated alkanes, with 27 the most abundant; C16:0 WE 40-48 even; secondary alcohols between WE; α-(ω-	Beeswax mixed with fatty materials.

						1) diols, WE 20:0; Unsaturated OL 32:1, 34:1; OL 12-19 also present; alkenes 23:1 and 31:1; very little saturated MAGs and DAGs; good range of FA from $C_{8:0}$-$C_{24:0}$, but not great abundance; two $C_{18:1}$ isomers; branched and straight chain $C_{15:0}$; two ω-2 hydroxies; diacid	Alkanes all attributable to beeswax, and higher plant OLs
5486	18.88	Fine Urfirnis sauceboat, body sherd	9		2	Large abundance of sterols; abundant β- sitosterol*, Δ^5 avenasterol*, and stigmasterol; small amount of cholesterol, campesterol, poriferasta-7, 25-dienol; cycloeucalenol, cycloartenol, or oleana-11,13(18)-diene; cyclolaudenol or 24-methylene-cyclocartenol; small amount of saturated MAGs; very small range of alkanes 20, 21, 22, 24; some alkenes and branched alkanes; OL 20, 22, 24; OL 14-16, 18 also present; OL 18 the most abundant, but very low abundance overall of OLs and alkanes	Unique plant-based residue with diverse sterol profile. Alkanol profile suggests only one to two plants. Probably a vegetable oil.
5489	8.0	Medium coarse base with sooting?	9		2	no sterols; only $C_{16:0}$, $C_{18:0}$, and $C_{18:1}$ FA; small amount of saturated MAGs; small range of alkanes (20-25), peaking at 22; two alkenes; small range of OLs 12-16, 18, 20; OL18 is the most abundant; minor branching of alkanes and OLs	Indeterminate residue.
5510	9.3	Coarse bowl with inturned rim, rim sherd	9		2	β-sitosterol; very small range of saturated FA $C_{14:0}$, $C_{16:0}$, $C_{18:0}$; small amount of saturated MAGs; alkanes 20-31, with 22 the most abundant; some branched alkanes and alkanols; smaller range of plant OLs (20, 22, 24); OL 13-19 present (except 17); OL 18 is the most abundant	Plant residue with some microbial degradation.
5665	48.7	Coarse body sherd; vessel with large pode	9		2	Cholesterol, 7-ketocholesterol, and possibly another cholesterol derivative; 15-hydroxy-7-oxo-DHA; FA range: $C_{6:0}$,$C_{9:0}$, $C_{12:0}$, $C_{14:0}$-$C_{18:0}$; straight chain and branched $C_{15:0}$ and $C_{17:0}$; $C_{16:1}$ and two positional	Mixture of pine resin, plant wax, plant/fish ($C_{16:1}$) and meat with microbial degradation. Multiple

						isomers of $C_{18:1}$; small amount of mostly saturated MAGs and 18:1 MAG; traces of saturated DAGs; odd-favored alkanes with 29 and 31 highest; branched alkanols and alkanes; OL 20-32 with OL 20 the most abundant; OL 12-19 also present; OL 18 is the most abundant OL	plants present from the range of alkanols.
5905	26.8	Medium coarse Urfirnis footed base	9		3	Cholesterol, Δ^5 avenasterol, and β-sitosterol; saturated MAGs; FA range $C_{12:0}$-$C_{18:0}$ (except $C_{17:0}$); $C_{18:1}$ in low abundance; alkanes 19-27, peaking at 22; some branched alkanes; alkenes 23:1 and 25:1; OL 20 and 22; OL 13-18 also present; OL 18 the highest; some secondary alcohols	Mixture of animal and small range of plants with minor microbial degradation. Small range of plants represented by OLs.
5920	14.5	Coarse body sherd	9		3	Cholesterol and β-sitosterol; small range and amount of FA ($C_{14:0}$, $C_{16:0}$, $C_{18:0}$); small amount of $C_{18:1}$; trace amounts of saturated MAGs; some branched alkanols and alkanes; alkenes; small range of OL 22, 24, 26 in very small amounts; OL 13-18 with OL 18 the most abundant alkanol; alkane 20 and 23 most abundant; alkanes range from 18-27, 29, 31	Degraded plant and animal residue. Small alkanol range suggests one plant source.
6682	915.1	Coarse base sherd	11	5a		Very abundant $C_{16:0}$, significant amount of $C_{24:0}$, odd favored alkanes dominated by 27 and 29, low even alkanes, very abundant OL 24, 26, 28, but all even OLs from 24-34, OLs make up 510.9 µg/g of the TLE; WE 40-46 even $C_{16:0}$ and WE $C_{20:0}$, very abundant WE, secondary alcohols amongst the WE, numerous alkenes; α-(ω-1) diols, most with branching; unsaturated OLs 28:1, 32:1, 34:1 a single MAG $C_{14:0}$; FA saturated $C_{12:0}$-$C_{24:0}$ (except $C_{23:0}$); $C_{14:1}$, $C_{15:1}$, $C_{16:1}$ (two isomers) and $C_{18:1}$ (two isomers); near total hydrolysis of TAGS and of WE	Complex mixture dominated by beeswax, but mixed with plants ($C_{16:1}$ and $C_{18:1}$) and other degraded fatty substances.

						large abundance of ketones: 16-hentriacontanone, 16-pentacosanone, 16-hexacosanone, nonacosan-25-one, 16-heptacosanone, 16-octacosanone, 16-nonacosanone, 15-nonacosanone; some branched alkanols; OL 12-22 also present in low abundance; diacids 4-8,11,14; odd diacids lower than even ones in the N, but low abundance; previously bound because in the N glycolic acid, 2-furoic acid, β-lactic acid, 5-oxy-valeric acid; no sterols	
6683	596.7	Medium coarse base	11	5a		odd alkanes 27, 29, 31, massive OLs 28, 30, 26, 24, 32, and some OL34, 335 ug/g of OLs in the TLE, massive $C_{16:0}$ and large $C_{24:0}$, secondary alcohols amongst the WE, abundant WE $C_{16:0}$ 40-48, numerous α-(ω-1) diols and α-ω diols; OL 32:1; alkenes, many of which are branched; wide range of FA from $C_{8:0}$-$C_{32:0}$ (except $C_{25:0}$-$C_{29:0}$ odd); three $C_{18:1}$ isomers, two $C_{16:1}$ isomers, and $C_{18:2}$; branched $C_{15:0}$; small amount of saturated MAGs/DAGs (1% of TLE); 1,2 diol nonacosane; OL 12-19 also; ketones K29 and 31; β-sitosterol; WE C20:0; diacids 6, 8, 10, 11; possibly dihydroxy or oxo FA in TLE	Beeswax mixed with plants. $C_{18:2}$ and $C_{16:1}$ indicate plants or fish. Good preservation.
7056	433.9	Coarse, short and squat jar, body sherd	11a	5		Abundant odd dominated alkanes 27>29>31>25>33; massive even alkanols 30>28>32>26>24>34, abundant $C_{16:0}$ and $C_{24:0}$; $C_{16:0}$ WE 40-48 even; also WE 20:0 and 18:0 FA components; secondary alcohols amongst the WE; alkenes 23:1 and 33:1; unsaturated OL 20:1, 22:1, and 34:1; abundant α-(ω-1) diols and α-ω diols; some branched alkanes and many branched alkanols; small amount of cholesterol and β-sitosterol;	Large amount of residue attributed to beeswax with at least a small plant, meat, and pine resin component with post-deposition microbial degradation, especially of the fatty substances. Excellent preservation, as indicated by the small FA and glycerol in the TLE.

						many branched FA, including $C_{15:0}$ and $C_{17:0}$; two isomers of $C_{18:1}$ and two isomers of $C_{16:1}$, but total unsaturated FA are low; full range of FA from $C_{7:0}$-$C_{24:0}$ (except $C_{19:0}$); low abundance and range of saturated MAGs; DHA; diacids 1,14 and 1,16; 2-decanone; valeric acid or succinic acid; 4-hydroxybutryic acid	
7063	25.4	Medium coarse, ovoid jar, yellow-mottled, body sherd	11a	5		Small amount of cholesterol; saturated MAGs; tight range of FA $C_{12:0}$-$C_{18:0}$ even and $C_{18:1}$; one possible WE with $C_{9:0}$ fatty acid component; numerous secondary alcohols; full range of alkanes 19-33, peaking at 22; OL 20-32 even dominated; OL 12-19 also present; OL 16 and then OL 18 the most abundant; many branched alkanols; unidentified compounds with m/z 73, 117, 131, 175	Mixture of meat and highly degraded plant wax from more than one plant.
7064 A	12.3	Medium coarse body?	11a	5		Very small amount of saturated MAGs; small range saturated FA $C_{14:0}$, $C_{16:0}$-$C_{18:0}$; some branched alkanes and alkanols; alkanes 19-27, 29, and 31 present, peaking at 22; OL 20-30 with OL 20 as the highest; OL13-18 also present; OL 18 the most abundant; no sterols	Indeterminate residue.
7064 B	9.9	Medium coarse body?	11a	5		β-sitosterol, Δ^5 avenasterol, and stigmasterol; small range of FA $C_{12:0}$-$C_{18:0}$; small amount of $C_{18:1}$; saturated MAGs; minor branched alkanols and alkanes; large range of alkanes from 19-26, 27, 29, 31, peaking at 22; OL 22, 24, 28 in low abundance; alkenes; OL 14-16 and 18 also present; OL 18 is the most abundant OL	Small amount of degraded plant residue.
7078	29.3	Fine ware body sherd	11a	5		Cholesterol and β-sitosterol; FA $C_{14:0}$-$C_{18:0}$ even; saturated MAGs and DAGs; alkanes range from 20-33, but peak 24-27; some branched alkanes; OL 22-28 even;	Mixture of plant wax and animal.

						OL 14, 16, 18 also present; alkanols in very low abundance	
7183	<5.0	Coarse ware, base sherd	11	5b		Not assessed	Interpretably empty.
7184	27.0	Medium coarse footed base and body	11	5b		Minute amount of β-sitosterol and cholesterol; saturated MAGs; FA $C_{12:0}$-$C_{18:0}$ and $C_{20:0}$; small amount of $C_{18:1}$; branched $C_{15:0}$ and $C_{17:0}$; alkanes 19-27, 29, 31, peaking at 22; alkenes 21:1 and 23:1; OL 20-26 even; OL 12-16, 18 preset; OL 18 the most abundant OL; glycerol in TLE; adipic acid; secondary alcohols	Mixture of plants and meat with some microbial degradation and decent preservation.
7219	23.5	Fine ware body sherd with gray fabric	11a		3b	β-sitosterol; FA $C_{12:0}$-$C_{18:0}$ even; saturated MAGs; alkanes 20-33, with alkane 24 and 26 as most abundant; some branched alkanes; OLs 22, 24, 26 in small amounts in the TLE only; OL 13-18, 20 also present; OL 18 the most abundant; OLs in low abundance	Plant residue, including epicuticular wax.
7370	7.3	Fine Urfirnis sauceboat, pedestaled base	9		1	Tiny amount of β-sitosterol and cholesterol; low abundance and small range of alkanes and alkanols; some saturated MAGs and DAGs; low abundance of FA $C_{14:0}$-$C_{18:0}$ even; no branching	Very little residue. Mixture of plant and meat?
7489	15.6	Coarse body sherd	9		1	Trace cholesterol and 15-hydroxy-7-oxo-DHA; odd dominated alkanes from 21-33 with 23 as the most abundant; alkenes 21:1 and 23:1; trace WE with $C_{14:0}$ and $C_{16:0}$ components; OL 20-30 even with no clear dominant OL; OL 14-19 also present; OL 18 is the most abundant; some branching of alkanes and alkanols; small range and low abundance of FA $C_{13:0}$-$C_{18:0}$ (except $C_{17:0}$); some $C_{18:1}$; small amount of saturated MAGs	Mixture of pine resin, meat, and plant epicuticular wax from more than one plant.

7538	< 5.0	Medium coarse pithos neck sherd	9		1	Not assessed.	Interpretably empty.
7599	133.4	Medium coarse bowl with incurving rim, body sherd	9		1	cholesterol, β-sitosterol, Δ^5 avenasterol; one 16:0 WE; one α-ω diol; very small amount of saturated DAGs; fair amount of mostly saturated MAGs and MAG 18:1; FA $C_{6:0}$, $C_{8:0}$-$C_{20:0}$ (except $C_{11:0}$ and $C_{19:0}$); some $C_{16:1}$; some branched OLs and many branched alkanes; several secondary alcohols; OL 20-30 even; OL 13-19 also present; OL 18 is the most abundant OL; alkanes 19-33, peaking at 24;	Mixture of meat, plant epicuticular wax, and other plant residue with microbial degradation. Multiple plant sources present. $C_{16:1}$ could originate from plant or fish sources.
7608	25.9	Coarse jar, body sherd	9		1	Trace amounts of cholesterol and stigmasterol; alkanes 18-33, peaking at 22; alkenes 21:1 and 23:1; even OL 20-26 with no clear dominant one; OL 14-19 also present; OL 18 is the most abundant OL; branched alkanes and alkanols; saturated MAGs; tiny amount of DAGs; FA $C_{7:0}$-$C_{18:0}$; $C_{18:1}$ in two isomers; straight and branched chain $C_{15:0}$ and $C_{17:0}$; two secondary alcohols; small amount of WE; K30 possibly	Mixture of plant and meat with microbial degradation, especially of the fatty substances.
7623	373.6	Coarse flared rim/body sherd	9		1	Significant amount of FA (70% of TLE); $C_{12:0}$-$C_{20:0}$ (except $C_{19:0}$); heavily saturated; branched $C_{15:0}$ and $C_{17:0}$; huge amount of $C_{16:0}$ and $C_{18:0}$, including underivatized $C_{18:0}$; small amount of two $C_{18:1}$ isomers; large amount of saturated MAGs and DAGs; 15-hydroxy-7-oxo-DHA and DHA methyl ester*; cholesterol, 7-ketocholesterol, and cholesta-3,5-dien-7-one; β-sitosterol and stigmastanol minute amount of WE 16:0 42 and 44 and C18:0 FA component; odd-dominated alkanes with 29 and 31 the highest; some branched; bacterial alkanes 18-19; alkane	Complex mixture of pine resin, meat, and plant products, including epicuticular wax with microbial degradation. Multiple plants represented from the range of OLs and alkanes.

						19 the highest of all alkanes; series of short chain alkenes; even dominated OL from 22-30; many branched OLs; OLs 12-20 also present with OL 18 most abundant; straight chain and branched 1,2 diols; K31 and K17	
7684	<5.0	Coarse ware, ovoid jar, yellow-mottled ware, base sherd	9	4		Not assessed.	Interpretably empty.
7760	106.1	Coarse body sherd	9		1	cholesterol and β-sitosterol; DHA and 15-hydroxy-7-oxo-DHA; MAGs/DAGs abundant at 12.5% of residue; saturated DAGs only; MAGs are saturated and unsaturated with $C_{15:1}$ and $C_{18:1}$; decent range of FA from $C_{6:0}$-$C_{20:0}$ (except $C_{10:0}$ and $C_{19:0}$); two positional isomers of $C_{18:1}$; branched and straight chain $C_{15:0}$ and $C_{17:0}$; somewhat odd-favored alkanes; alkane 23 is the highest; full range alkanes from 18-33; alkenes 19:1, 20:1, 21:1, 23:1; even dominated alkanols from OL 20-34; OL 12-19 also present; long chain secondary alkanols; OL 18 the most abundant; numerous branched alkanols and alkanes	Mixture of plant epicuticular wax and meat with pine resin with microbial breakdown. More than one plant source.
8020	163.6	Bulbous, medium coarse jar with hole, body sherd	9		1	Odd-dominated alkanes with 31 the most abundant; $C_{16:0}$ WE 40-48; abundant $C_{16:0}$; OL 26-34 even, dominated by OL28; wide range of FA $C_{8:0}$-$C_{10:0}$, $C_{12:0}$-$C_{18:0}$, $C_{22:0}$-$C_{30:0}$ (except $C_{21:0}$, $C_{23:0}$, $C_{29:0}$); MAGs/DAGs minimal and all saturated; $C_{18:1}$ coeluted with an unidentified compound; DHA; 7,15-dihydroxy DHA; β-sitosterol, Δ^5 avenasterol, and cholesterol; alkanes range from 18-33;	Possibly degraded beeswax mixed with pine resin, animal, and plants. Multiple fatty substances are present. Some microbial degradation.

						some secondary alcohols; OL 13-20 also present in low abundance; branched OLs	
8072	31.6	Medium coarse Urfirnis? Body sherd	9		2	Small amount of saturated DAGs and MAGs; $C_{6:0}$, $C_{8:0}$, $C_{9:0}$, $C_{15:0}$, and $C_{12:0}$-$C_{18:0}$ even; small amount of $C_{18:1}$; odd-dominated alkanes, dominated by 31 and 29; even OL 24-28; OL 12-16, 18 also present; OL 18 is the most abundant OL; some branched OLs; no sterols	Plant epicuticular wax? Good preservation from small fatty acids.
8092	104.8	Medium coarse shallow bowl, rim/body sherd	9		1	β-sitosterol and cholesterol; abundant FA ranging from $C_{12:0}$-$C_{24:0}$ (except odd $C_{19:0}$-$C_{23:0}$); branched $C_{15:0}$; two $C_{18:1}$ isomers; good range of MAGs (all saturated); very small amount of saturated DAGs; small amount of two WE; wide range of alkanes 18-32, 33, with peaks at 20 and 23; three alkenes; numerous branched alkanes and alkanols; some secondary alcohols; OL 20-32 even dominated; OL 12-18 also present; OL 16 and 18 the most abundant	Mixture of plant wax and animal products with microbial degradation. Multiple plants present.
8096	105.6	Coarse, short and squat jar, body sherd	9	5a		Cholesterol, 7-ketocholesterol, and cholesta-3,5-dien-7-one; β-sitosterol, Δ^5 avenasterol, and stigmasterol; abundant WE (13.11 μg/g) belonging to $C_{16:0}$, $C_{16:1}$, $C_{13:0}$, $C_{12:0}$, and $C_{14:0}$ FA; wide range of FA $C_{8:0}$-$C_{26:0}$ even and $C_{9:0}$-$C_{17:0}$ odd; branched $C_{15:0}$ and $C_{17:0}$; unsaturated FA in decent amount $C_{14:1}$, $C_{16:1}$, $C_{17:1}$, and $C_{18:1}$; abundant saturated DAGs/MAGs; small amount of MAG C18:1; many branched OLs and some branched alkanes; even favored OLs from 20-30; OL 20:1, 22:1, 24:1; OL 13-19 also present; most abundant OL 18; alkanes range from 18-33, but peak at alkane 21; alkenes 21:1 and 23:1	Complex mixture of animal products, strong plant epicuticular wax component, and plant/fish, with microbial degradation. Wide range of OLs suggests more than one plant represented. Wide range of FA suggests multiple fatty sources.
8141	55.1	Coarse handle	9		1	Alkanes odd-dominated with alkane 31 most abundant; OLs even-dominated from 22-34, with OL 30 most abundant; OL 15-21 also present (except OL 17) and	Strongly plant-based residue with minor animal contribution. Alkane and alkanol sequence look like

						peak at OL 18; β-sitosterol, stigmasterol, and trace amount of cholesterol, which could be from plants; saturated DAGs/MAGs; some branched alkanes; FA $C_{12:0}$-$C_{24:0}$ even saturated and small amount of $C_{18:1}$; $C_{16:0}$ most abundant FA	degraded beeswax, but no WE. $C_{18:1}$ could be plant or animal derived.
8156	< 5.0	Fine ware with grey fabric, body sherd	9		1	Not assessed.	Interpretably empty.
8428	49.0	Small medium coarse bowl with inturned rim, burnished, rim sherd	9	4		Small amount of cholesterol and Δ^5-avenasterol; $C_{14:0}$-$C_{18:0}$ even saturated; some $C_{18:1}$; minor saturated and unsaturated MAGs; three very abundant secondary alcohols, which make up 1/3 of total alkanols; OL 20-28 even in low abundance; OL 13-16, 18, 21 also present; OL 18 most abundant OL; a narrower range of alkanes: even-dominated from 25-31, but in low abundance; alkane 22 is the most abundant; neutral is unusable	Animal based residue with epicuticular plant wax from more than one plant source.
8506	19.3	Coarse jar, rope decoration, body sherd	9	4		Cholesterol, 7-ketocholesterol; FA $C_{6:0}$, $C_{9:0}$, $C_{12:0}$-$C_{18:0}$; branched $C_{15:0}$; small amount of $C_{16:1}$; $C_{18:1}$ inferred from trace $C_{9:0}$; two WE $C_{16:0}$; α-ω diol?; saturated MAGs; alkanes from 18-31; alkane 20 and 22 the most abundant; alkenes 19:1, 20:1, 21:1, 23:1; branched alkanes and OLs; OL 22-28 even in low abundance; OL 12-19 also present; OL18 is the most abundant; two secondary alcohols	Meat and plant wax with microbial degradation. $C_{16:1}$ could be plant or fish derived.
8849	15.5	Coarse body sherd, yellow-mottled, Sooting exterior	11	4		Cholesterol, β-sitosterol, stigmasterol, and Δ^5 avenasterol; DHA; small amount saturated MAGs; wide range of FA $C_{6:0}$-$C_{9:0}$, $C_{12:0}$-$C_{26:0}$ even, $C_{11:0}$, $C_{15:0}$, $C_{17:0}$; branched $C_{15:0}$ and $C_{17:0}$; unsaturated FA at 4% ($C_{18:2}$, $C_{18:1}$, and two $C_{16:1}$ isomers); succinic diacid; alkanes and alkanols are branched, but not extensively; alkanes full range and peak 23 and 24; OL 20-30 even	Mixture of plant wax, animal, and plant/fish ($C_{18:2}$ and $C_{16:1}$) with pine resin with some microbial degradation. More than one plant represented. Multiple fatty sources. Good preservation. Wide range of

						dominated; OL 13-19 present with OL 18 the most abundant; laevulic acid	FA and quite diverse for how little residue
10421	19.0	Medium coarse base	Balk A	5b/ 4		cholesterol and β-sitosterol; somewhat odd-dominated alkanes with 31 as the most abundant; alkanes 20 and 22 also in large amounts; OLS 22-32 with OL 28 slightly larger than the rest; OL 16, 18, 19 also present; OL 18 the most abundant; many branched alkanols and alkanes; some alkenes; unsaturated OL 20:1; small range of FA $C_{14:0}$-$C_{18:0}$ even, $C_{15:0}$, and $C_{18:1}$; MAGs saturated	Mixture of plants and meat with microbial degradation.
11277	46.9	Coarse jar? body sherd	12	4		Range of sterols: cholesterol and campesterol in very small amounts; stigmasterol, β-sitosterol, Δ^5-avenasterol, poriferasta-7, 25-dienol, possibly dihydrolanosterol?; DHA and 15-hydroxy-7-oxo-DHA; cycloeucalenol, cycloartenol, or oleana-11,13(18)-diene; fairly wide range of FA: FA $C_{6:0}$-$C_{20:0}$, except $C_{11:0}$ and $C_{19:0}$; branched C15:0, $C_{16:0}$,$C_{17:0}$; some branched and unsaturated alkanes; alkanes 18-32, peaking at 22; OL 22-30 even, but in low abundance; OL 12-18, 21 also present; OL 18 the most abundant; diols; WE $C_{14:0}$ and $C_{16:0}$ FA components; MAG C18:1 in 1- mono and 2-mono position are the most abundant MAGs; saturated MAGs; saturated and unsaturated DAGs, but tiny amount;	Unique mixture of pine resin and plant sterols with epicuticular wax from more than one plant. Some $C_{18:1}$ from DAG and inferred from $C_{9:0}$, but in low abundance. $C_{18:1}$ could originate from plant or animal. Cholesterol could originate from plant(s), because it is in such low amounts. Some microbial degradation of the fatty substances.
11309	95.9	Coarse base/body of handled open vessel with flaring rim	8	4	1	β-sitosterol and cholesterol; FA even $C_{14:0}$-$C_{22:0}$, $C_{15:0}$ and $C_{17:0}$; branched $C_{16:0}$ and $C_{17:0}$; $C_{18:1}$; saturated DAGs small amount; saturated MAGs and MAG $C_{15:1}$; branched alkanes and alkanols; OL 20-30; OL 12-19 also present; OL 18 is the most abundant; alkanes 18-33, peaking at 24; alkanes 21:1 and 23:1; laevulic acid; adipic acid; one branched a-ω diol	Mixture of plant and meat products with microbial degradation. More than one plant present.

12461	39.0	Medium coarse, flared rim sherd, Urfirnis?	Balk A	4		Abundant and diverse sterols at 12.43 µg/g of the TLE, dominated by β-sitosterol; Δ^5avenasterol; unidentified sterol; poriferasta-7,25-dienol; cyclolaudenol or 24-methylene cycloartenol; stigmasterol; unidentified stigmasterol relative; campesterol; cycloeucalenol, cycloartenol, or oleana-11,13(18)-diene; small amount of cholesterol; cholesterol is the least abundant sterol; Small amount of saturated MAGs/DAGs; small range of FA $C_{14:0}$-$C_{20:0}$ even and $C_{18:1}$; alkane 29 and 31 dominate; alkanes 20, 23-27 also present; OL 20, 22, 24 present in low amounts; OL 12-18 also present; OL 18 the most abundant OL; minor branching in alkanes and OLs	Heavily plant-based residue. Cholesterol could be of plant origin. Excellent preservation. OLs suggest limited plant sources. Unique sterol profile suggests vegetable oil.
14750	112.9	Medium coarse body sherd	Balk A	4		Cholesterol and 7-ketocholesterol; small quantity of Δ^5-avenasterol; 15-hydroxy-7oxo-DHA; WE $C_{14:0}$ and $C_{16:0}$ FA component; not abundant MAGs/DAGs and all are saturated; decent range of FA $C_{9:0}$, $C_{12:0}$-$C_{18:0}$, $C_{24:0}$; $C_{15:0}$ and $C_{17:0}$ branched and straight chain; some $C_{16:1}$ (but coelute); fair amount of $C_{24:0}$; abundant and wide range of alkanes from 18-33, peaking at 24, and making up 49% of the TLE; numerous branched alkanes; even-dominated from OL 20-30; OL 13-19 also present; OL 18 is the most abundant; some branched OLs	Mixture of meat, pine resin, and plant wax? More than one plant represented with heavy microbial degradation.
15787	13.6	Medium coarse small bowl, rim/body sherd	8	3		Small amount of cholesterol (0.08 µg/g); small range of saturated FA $C_{14:0}$-$C_{18:0}$ even and $C_{15:0}$; some $C_{18:1}$; small amount of saturated MAGs; alkane 20; OL 20-24; OL 14-16, 18, 19 also present; OL 18 is the most abundant; no branched alkanes and alkanols; neutral is unusable	Small amount of animal and plant residue from only 1-2 plants with slight microbial degradation.

Abbreviations and Symbols:
*= compound found in the TLE or N blank, but in a small amount relative to residue, so the amount was just subtracted out
N= neutral component of the residue
TLE=total lipid extract
OL= alkanol
WE= wax ester
DHA= Dehydroabietic acid
FA= fatty acid
K=ketone
MAG=monoacylglyceride
DAG= diacylglyceride

VITA

Rachel was born in Texas to Pam and Paul Vykukal. She later relocated with her family to Spartanburg, South Carolina where she attended primary and secondary school. She graduated Salutatorian of her high school class and went on to pursue an undergraduate education at the College of Charleston in Charleston, South Carolina. Rachel graduated *summa cum laude* in 2005 with a Bachelor of Arts in Studio Art with an Arts Management minor and a Bachelor of Science in Anthropology. She returned to the College of Charleston in 2008-2009 to complete a third undergraduate degree, a Classics focused *Artium Baccalaureatus* in Anthropology, before moving to Tennessee to pursue graduate school. She was awarded separate graduate fellowships from the University of Tennessee-Knoxville to pursue both M.A. and Ph.D. degrees. She focused her M.A. research and subsequent thesis on Royal Purple dye production in mainland Greece in the Bronze Age. She graduated from the University of Tennessee-Knoxville with a Master of Arts in Anthropology with a concentration in Mediterranean Archaeology in 2011. She shortly thereafter matriculated into the Ph.D. program in Anthropology at the same university. She continued to pursue Mediterranean archaeology, incorporating archaeological chemistry and organic residue analysis into scholarship of the region. Rachel graduated in August 2018 with a Ph.D. in Anthropology.